2007

Use of ground clay brick as a supplementary cementitious material in concrete hydration characteristics, mechanical properties, and ASR durability

Fatih Bektas
Iowa State University

Recommended Citation

Bektas, Fatih, "Use of ground clay brick as a supplementary cementitious material in concrete hydration characteristics, mechanical properties, and ASR durability" (2007). *Retrospective Theses and Dissertations*. 97.
https://lib.dr.iastate.edu/rtd/97

Use of ground clay brick as a supplementary cementitious material in concrete -hydration characteristics, mechanical properties, and ASR durability

by

Fatih Bektas

A dissertation submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Civil Engineering (Civil Engineering Materials)

Program of Study Committee:
Halil Ceylan, Co-major Professor
Kejin Wang, Co-major Professor
Max L. Porter
Paul G. Spry
R. Christopher Williams

Iowa State University

Ames, Iowa

2007

CONTENTS

Acknowledgements

I would like to thank my supervisors, Dr. Kejin Wang, for her perceptiveness, insightful suggestions and continuous supervision during the study; and Dr. Halil Ceylan, for his invaluable help and open-minded discussions.

I am grateful to my former supervisor, Dr. Lutfullah Turanli, who never spared his support despite the thousands of miles between us.

Gratitude is also extended to my committee members, Dr. Max Porter, Dr. Paul Spry, and Dr. Chris Williams, who have provide me with great encouragement in my research and constructive comments on this dissertation.

I also wish to thank the personnel of the PCC Lab of CP Tech Center (Ames, IA) for their helps. Particularly, I would like to express my sincere appreciation to our lab manager Bob Steffes: without his innovative approaches this work could have never been possible.

Thanks to real friends who make the academic life more bearable.

I am always in debt to my family for their endless understanding: many thanks to them.

Finally, a big 'thank-you' for my wife, Basak, who has the courage to walk with me.

Fatih Bektas

Abstract

The work presented in this Dissertation involves an effort to broaden sustainable applications (e.g., recycling waste material in concrete, reducing CO_2 emissions in cement production) in construction industry. Finely ground brick obtained from demolished masonry was evaluated as a pozzolanic cementitious material in concrete. The hydration characteristics of this material and its effects on cement paste, mortar, and concrete mechanical properties, particularly on alkali-silica reaction deterioration was studied.

Ground clay brick (GCB) was produced in the laboratory employing a ball mill. Chemical and physical properties comply with the designated ASTM standard that defines pozzolanic materials.

The results of the study on cementitious pastes where cement was replaced by GCB up to 25% showed that the effect on water demand for normal consistency and time of setting was insignificant; the temperature rise during hydration was reduced; and the calcium hydroxide production was decreased. The testing of mechanical properties of concrete containing up to 25% GCB demonstrated that the values were low at early age but higher or comparable at later ages when compared to the control and the resistance to chloride penetration was improved. These results suggested a pozzolanic activity provided by finely ground brick.

The results from the accelerated ASR testing of mortar mixtures showed that the expansions were significantly reduced and associated deterioration on the compressive and flexural strengths was limited with the use of GCB. The concrete tests confirmed the mortar testing that the expansion was effectively suppressed and the mechanical

properties of a highly alkali-silica reactive mixture could be conserved when cement was replaced by GCB at 15 and 25% loading. Subsequent microstructural study revealed out the quantitatively low gel formation in the presence of GCB and further suggested a modification in gel chemistry by GCB.

The reported experimental study demonstarted that GCB shows pozzolanic behavior similar to other conventional materials (e.g., fly ash, calcined clay, or natural pozzolan), and it can be used as a supplementary cementitious material in concrete applications. Use of GCB in concrete can effectively reduce the alkali-silica reaction deterioration in concrete.

Chapter 1

Introduction

1.1. Background

Concrete is one of the oldest and the most widely used construction material in today's world. It is easily obtainable, relatively cheap, strong, and durable. On the other hand, the concrete industry is one of the major consumers of the natural resources. The annual concrete production is estimated as 11 billion metric tons[1][♠]: 70–75% of the number is aggregate (mostly natural rock); 15% is water; and 10–15% is cementitious binder. The majority of the cementitious binder used in concrete is based on portland cement clinker which is an energy-intensive process. Global cement production was 2.3 billion tons in 2005 which is almost four times the number in 1970[2]. One ton of cement production is responsible for one ton of CO_2 emission: half of the CO_2 is from the chemical process of clinker production, 40% from burning fuel, and the remaining 10% is split between electricity use and transportation[3]. According to the recent data, cement clinker production is the largest CO_2 source among industrial processes: it contributes about 4% of global total CO_2 emissions from fuel use and industrial activities[4].

The impact of cement/concrete industry on environment is significant. With the growing environmental concerns, public awareness has been increased together with its pressure on the industry. More and more sustainable approaches are being researched and implemented. Recycling waste material or industrial by-products in concrete is one of the well-known applications. Instead of using natural resources, recycled aggregate obtained from old concrete and demolished masonry are used in concrete, even in structural applications. Additionally, industrial by-products such as fly ash, ground granulated blast furnace slag, and ground waste clay brick are replaced by more energy-intensive portland

---

[♠] Each chapter has its own reference listing at the end.

cement. These supplementary cementitious materials also improve concrete durability such as resistance to sulfate attack and alkali-silica reaction.

1.2. Objective of Dissertation

The objective of the present work is to study the suitability of finely ground waste/recycled clay brick as a supplementary cementitious material in concrete. Fired clay bricks may possess pozzolanic property due to their high silica and alumina content and some are known to increase mortar durability, resistance to sulfate attack and alkali-silica reaction (ASR). It is necessary to achieve a better understanding of the ground clay brick in concrete application when used as a partial cement replacement. An experimental program was designed to investigate the effect of ground clay brick on hydration characteristics and concrete mechanical properties. The effect of ground clay brick on ASR associated deterioration in concrete was also analyzed as it is a major concern for concrete performance.

1.3. Dissertation Organization

This dissertation is divided into seven chapters. Chapter 1 provides an introduction and background information on this dissertation.

Chapter 2 includes a brief literature review of the use of recycled clay brick in cement-based materials. The review focuses on the use of the material as aggregate and as cementitious binder. In the literature review, a section on pozzolan-ASR relationship is added.

The main findings and results of the study are presented in the second part (Chapter 3–5). Each chapter comprises a paper that has been either published, submitted for publication, or ready for submission to peer reviewed journals. The papers are ordered in the thesis as follows:

Bektas F, Kejin W, Ceylan H. *Use of Ground Clay Brick as a Pozzolanic Material in Concrete*. Will be submitted to Building and Environement (Elsevier).

Chapter 3 covers the effect of ground brick addition (up to 25%) on the hydration characteristics of cementitious paste—water demand for normal consistency, initial and final time of setting, temperature rise during hydration, and calcium hydroxide consumption. Effect on the mechanical properties and chloride ion penetration of concrete mixture is also given.

Bektas F, Turanli L, Kejin W, Ceylan H. *Comparative Performance of Ground Clay Brick in Alkali-Silica Reaction*. Accepted for publication in Journal of Materials in Civil Engineering (ASCE).

Chapter 4 provides the results for the use of ground clay brick in mortar up to 35% cement replacement. The ASR performance of ground brick in comparison to conventional supplementary cementitious materials, namely low-calcium fly ash and volcanic origin natural pozzolan was studied. Effects of ground clay brick addition on ASR-related damage (i.e., expansion and strength loss) are presented.

Bektas F, Kejin W, Ceylan H. *Control of ASR Damage on Mechanical Properties of Concrete Using Ground Clay Brick*. Will be submitted to Cement and Concrete Composites (Elsevier).

Chapter 5 includes the findings of the study concerning the use of ground clay brick to prevent ASR damage in concrete mixture. The performance is compared to high-calcium fly ash (Class C) in concrete mixtures containing up to 25% pozzolan replacement of cement. The effects on concrete expansion and mechanical properties were studied.

Finally, Chapter 6 summarizes the major findings of the study and Chapter 7 gives recommendations for industrial applications and future research.

Appendix A includes the papers by the author that are submitted to journals. The research leading to these papers were performed during the PhD study but not included as part of the main thesis:

Bektas F, Kejin W, Ceylan H. *Effcets of Crushed Clay Brick on Mortar Durability*. Submitted to Construction and Building Materials (Elsevier).

Bektas F, Kejin W, Ceylan H. E*ffect of Portland Cement Fineness on ASTM C 1260 Expansion*. Submitted to Journal of Testing and Evaluation (ASTM).

Appendix B contains the pictures of the equipment used in the experimental study.

Appendix C includes a resume of the author.

References

[1] Mehta PK, Monteiro PJM. Concrete—Microstructure, Properties, and Materials. New York, NY: McGraw Hill, 2006.

[2] Taylor M, Tam C, Gielen D (2006). Energy efficiency and CO_2 emissions from the global cement industry. In: International Energy Agency - World Business Council for Sustainable Development WorkshopEnergy Efficiency and CO_2 Emission Reduction Potentials and Policies in the Cement Industry, Paris, France. (http://www.iea.org)

[3] World Business Council for Sustainable Development (WBSCD) (2005). The cement sustainability initiative progress report. (http://www.wbcsdcement.org)

[4] Netherlands Environmental Assessment Agency (2007). China now no. 1 in CO2 emissions; USA in second position. (http://www.mnp.nl)

Chapter 2

Literature Review: Recycling Clay Brick in Concrete

2.1. Terminology

In this dissertation, unless stated differently, the word ‘brick’ refers to ceramic masonry unit which is manufactured by firing clayey soil. ‘Recycled clay brick’ is defined as the waste material that can be obtained from demolished masonry or non-standard discarded products at the end of the manufacturing process. Additionally, ‘crushed brick’ is the form of this material in aggregate size (0.075–50 mm) and ‘ground brick’ is the form powdered to cement fineness. The particle sizes may overlap; but the purpose of the usage defines the material definition used in the study. (e.g., crushed brick and ground brick are both obtained from recycled clay brick.).

2.2. Sources of Recycled Brick

There are two major sources from which recycled clay brick can be obtained—construction and demolition waste, and clay brick/tile manufacturing plants.

Construction and demolition waste (CDW) includes the unwanted leftover material from any construction activity which can be new construction, renovation or demolition. In developed countries hundreds of million tons of CDW is generated annually. In the European Union the annual CDW production is 200 million tons and according to EPA annually 215 million tons of CDW s generated in the US.[1, 2] Although no accurate records exist for developing countries, the CDW amount could be expected high considering the massive infrastructural development in these countries. Fired clay products, particularly brick that can be recycled, correspond to significant amounts in CDW. In EU alone, masonry CDW may constitute 42–92% of a typical CDW mass and 30–80% of it is ceramic materials (brick and tile).[3]

The earliest fired bricks discovered so far dates back to 5000–4500 BC in Mesopotamia and they are still being manufactured and used in construction in the 21st century.[4] The manufacturing process has been refined over thousands years of experience, however, no technology is available to manufacture with hundred percent production efficiency. Despite the advanced technology used in nowadays clay brick manufacturing, t significant amounts of products are still discarded because they do not confirm the standards. Due to improper heating regimes these units may be overburned, underburned or distorted simply not fulfilling the standard requirements. In a typical hi-tech plant in the US the fired material discarded constitutes 3% of the total production.[5] In some European countries the figure is between 2–5%.[6] Higher percentages could be expected in low-efficiency plants mostly flocated in developing countries. For instance, from a single brick field in Bangladesh the material treated as waste or useless is 10–15% of the total stock.[7] High amounts of waste ceramic materials are also produced in other countries such as Brazil, Portugal, and Turkey.[8–10] Some of these rejected fired products are recycled to manufacturing process as raw material, and named as grog or clinker brick. However, the majority is dumped.

2.3. Recycled Brick as Concrete Aggregate

Recovery of brick units from masonry built with portland cement mortar is impractical for re-use since the bonding is too strong. Therefore, they are mostly crushed and used with the mortar impurity. A selective screening to obtain ceramic material is also possible. Obviously waste material from the manufacturing plants does not have this problem.

Today recycled brick is used as overlay material in tennis courts and tracking fields and as plant substrate. In civil engineering applications, it can be used in unbound systems such as drainage blankets, subbase in road construction, or fill material in embankments.

On the other hand, high-grade utilization, such as an ingredient in concrete or asphalt, is also possible.

Crushed brick aggregate in portland cement concrete is known to be used in Germany in 1860. Systematic investigations on the use of crushed brick aggregate dates back to 1928. However, the first significant practical application was after the Second World War in Germany where the cities were destroyed down to rubble. Approximately 11.5 million cubic meters of crushed brick aggregate were used to build 175,000 dwellings. The benefit was two-fold—removing the masonry rubble and meeting the aggregate demand for new construction.[11] The research on crushed brick aggregate has slowed down in the preceding decades. However, there is an increasing revisit in the first decade of the 21st century. The new motive is the sustainability: the construction industry is trying to reduce its impact on the environment by increasing recycling efforts. In addition, natural sources are being depleted and dumping is becoming costly in metro areas. For instance, Bangladesh lacks good quality rocks or boulders, and aggregate import is not economical, therefore, brick is intently produced for concrete aggregate.[7] Besides, in highly populated metro areas such as Hong Kong disposal landfills are limited and costly.[12]

Studies on brick aggregate in concrete generally involve the utilization of brick as coarse aggregate. Parent brick determines the characteristics of the concrete produced with crushed brick aggregate: (1) concrete unit weight decreases with increasing porosity; (2) water demand of the mixture increases with increasing water absorption of the brick aggregate, thus concrete strength reduces proportionally; and (3) concrete strength increases with increasing brick strength. Brick is porous in nature and the bulk density of brick aggregate falls somewhere in between normal-weight and light-weight aggregate which reduces the unit weight of the concrete produced from crushed brick aggregate. Replacing coarse aggregate with crushed brick Khaloo[13] was reported compressive strengths of 35.7–39.0 MPa at 28 days. Similarly, Mansur et al.[14] reported 40–70 MPa cube strengths with varying water-to-cement ratio crushed brick concrete. Tensile and flexural strengths have been found to increase for crushed brick concrete compared to normal aggregate. This increase may be attributed to improved bonding between brick aggregate and cement paste.[15] In contrast to other mechanical properties, modulus of elasticity of crushed brick concrete may be 30–50% lower compared to normal weight aggregate.[11,16]

Research on crushed brick as fine aggregate is relatively new. The reluctance in using it as fine aggregate was probably due to abnormally high water demand that might be caused by fine crushed brick. Recently, there have been several studies focusing on crushed brick fine aggregate. Debieb and Senai[17] have reported significant losses in compressive and flexural strengths. On the other hand, Khatib[18] have found comparable strength results with the control mixture containing normal fine aggregate and attributed this performance to the pozzolanic activity of very fine brick particles.

Surprisingly, research on durability performance of crushed brick aggregate is very limited in the literature. As a durability measure, water tightness (or water penetration) was investigated and found to be 50% higher than that of normal control concrete.[11] Padmini et al.[19] stated the rate of carbonation is higher. Bektas et al.[20], in their study with mortars incorporating crushed clay brick, found out that freeze-thaw resistance of mortars may increase crushed brick aggregate; however, the crushed brick aggregate is potentially ASR susceptible.

The studies to date have well established that concrete with adequate mechanical properties can be produced using crushed brick, particularly, as coarse aggregate. Further research is needed for durability aspects.

2.4. Recycled Brick as Pozzolan in Concrete

Pozzolanic materials, or pozzolans, (e.g., fly ash, silica fume, volcanic tuff, rice husk ash, metakaolin) are used in concrete as secondary binder together with portland cement. They reduce permeability and increase durability such as resistance to sulfate attack and ASR.

Pozzolan is defined in ACI 116R[21] as:

> "a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties."

When used in portland cement systems, pozzolanic materials react with portlandite (or calcium hydroxide), which is a reaction product of portland cement and water, and form secondary calcium-silicate-hydrate (C-S-H) gels. C-S-Hs are the main reaction products of cement and water interaction. They are the glue of concrete matrix: they occupy the voids between aggregate particles keeping the matrix together. Some of the water used in concrete mixture is not consumed by the cement hydration; therefore, voids left upon evaporation of the excess water. Secondary C-S-Hs formed during pozzolanic reaction grow into these extra voids, and refine concrete porosity. Improved internal structure is often a sign of increased durability.

Pozzolans have been known to construction industry since the ancient Greeks. The volcanic ash from Santorin Island in Aegean Sea was used in lime mortars to build durable structures.[21] The successors of the Greeks, the Romans, refined the practice and discovered new pozzolan sources one of which is brick powder. When the natural pozzolanic material was not available the Romans used powdered brick to mix with lime to obtain a strong, durable binding material. They used lime mortars containing crushed brick and brick powder in floors and foundations for water proofing purposes.[22] The Romans called this mortar as *cocciopesto*, which was inherited by the Ottomans as *horasan*. *Surkhi* and *homra* are the names used in India and in the Arabian Peninsula, respectively.

Some clay minerals possess pozzolanic property when thermally treated: upon heating interlayer water is removed, crystal structure is destroyed, and a new material with pozzolanic property is formed. Particularly, kaolin clay demonstrates high pozzolanic activity: kaolin looses its structural water around 600°C and metakaolin forms.[23] This new material is used as pozzolan in finely divided form in cement-based systems. Calcined kaolin, or metakaolin, has been successfully used in mass concrete applications in Brazil since 1965. The production cost was one third of the portland cement and it suppressed the potential alkali-aggregate reaction in several dams built with reactive aggregate.[24] Ambroise et al.[25] found that the most kaolin-rich and the mostly poorly crystallized clay shows the best pozzolanic activity. Kaolin is an essential ingredient of clay brick manufacturing due to its sintering behavior.[26] Therefore, kaolin-rich clay brick may show pozzolanic activity in finely divided form and has potential to be used as supplementary cementitious material. Wild et al.[27] achieved to produce pozzolan by heat treatment of Oxford clay used in brick production.

Research proved that finely ground clay brick has potential to be used as a pozzolanic material in cement-based systems. In their study with eight different clay bricks from Britain, Denmark, Lithuania, and Poland, Wild et al.[28] carried out chemical test for pozolanicity and compression test for strength development of mortar mixtures containing up to 30% ground brick as cement replacement; and concluded that all the ground brick types used in the study exhibit pozzolanicity. It has been also found that ground brick improves the resistane of mortar to aggressive environments. O'Farrell et al.[29,30] showed the resistance of mortar mixtures to sulfate attack is increased when ground brick is added to mixture; similarly, negative effects (expansion and strength reduction) of seawater on the mortar mixtures were mitigated. A recent study on mortars containing up 40% of waste clay ground brick demonstrated that the compressive strength is not negatively affected up to 20% replacement and resistances to chloride ion penetration and sulfate attack are increased.[8] Alkali-silica reaction resistance of mortar mixtures has also been found to improve with the ground brick addition.[6,31] On the other hand, research on ground brick using concrete mixtures are very limited and confined to compressive strength determination. In a comprehensive research project on the use of waste clay brick and tile material as partial cement replacement, ground brick is found to decrease early age strength but at later ages strength values of ground brick concrete reach or exceed that of control mixture; additionally, elastic modulus is slightly reduced at all ages.[6] Nonetheless, the research has proved that concrete mixtures with ground brick can be proportioned to have adequate mechanical properties.

2.5. Pozzolans and ASR

Since its initial discovery by Stanton in the early 1940s, ASR has been one of the major durability concerns of the construction industry. ASR is the chemical interaction of concrete alkalis and reactive siliceous aggregate. The reaction product is a gel (ASR gel), which absorbs water and swells. The swelling pressure is often sufficient to disturb the volumetric stability and cause cracking. Eliminating one of the elements of the reaction, namely reactive aggregate, alkali, water, is either impractical or uneconomical.

Use of pozzolans in concrete mixture is the most prudent method to eliminate ASR associated complications. Pozzolans are abundantly available and has been proven to be effective in ASR mitigation. Effectiveness of pozzolans depends on reactivity, amount, and size of aggregate, environmental conditions (temperature, moisture and presence of external alkali source), total alkali-content of concrete mixture, and type and amount of pozzolan used. In addition, for the same type of pozzolan, the effectiveness is highly related to physical and chemical characteristics. The desirable characteristics for reducing the expansion are (1) low alkali content, (2) high total acidic oxides (SiO_2 + Al_2O_3 + Fe_2O_3), and (3) high fraction of particles finer than 45 micron.[33] Different explanations have been proposed on how pozzolans mitigate ASR. The actual condition is certainly a combination of different mechanisms—dilution effect, pore- or grain-size refinement, consumption of $Ca(OH)_2$, and entrapment of alkalis.[♣]

Dilution effect is the very first and simple explanation. Blending agents act as a diluent, because they are less reactive than portland cement and liberate alkali at smaller rates. Xu et al.[34] partially replaced cement by powdered calcium carbonate, which was assumed chemically inert, and the deleterious expansion was less compared to that of the control. Some supplementary cementitious materials, however, may have higher soluble-alkali content than cement which proposes additional mechanisms should be involved.

As mentioned earlier, pozzolans cause pore-size and grain-size refinement leading to a dense and less permeable concrete. Consequently, ionic mobility in concrete is limited: migration of hydroxyl (OH^-) and alkali (Na^+, K^+) ions towards reactive aggregate particles is limited, thus, initiation of the deleterious reaction is restricted.[35,36]

Another mechanism is the consumption of $Ca(OH)_2$, thus, reduction of OH^- in pore solution. Negative effect of $Ca(OH)_2$ on alkali-silica expansion is known. Wang and Gillot claimed that $Ca(OH)_2$ acts as a buffer to maintain pH value in the pore solution. It releases hydroxyl ions which attach to siliceous aggregate and form negatively charged sites suitable for alkali ions. This is the formation of alkali-silica gel in the simplest term. Furthermore, calcium ions may exchange for alkali ions in the system and increase pore solution alkalinity.[37] Pozzolans lower the $Ca(OH)_2$ content of cement paste reducing its pH. Direct measurements of pore solutions using high-pressure technique have demonstrated how silica fume replacement reduced the hydroxyl ion concentration.[38] Shehata et al.[39] showed that the hydroxyl ion concentration in the pore solution of pastes having fly ash is reduced compared to that of pastes having no fly ash. Similarly, Ramlochan et al.[40] explained that hydroxyl ion concentration in the pore solution

---

[♣] The following part is rephrased from *Bektas F, Preventive Measures Against Alkali-Silica Reaction, MSc Thesis. METU: Ankara, Turkey, 2002.*

decreases as the amount of metakaolin in the paste increases. The effect of $Ca(OH)_2$ removal from the system on ASR also seems to be correlated with calcium ions. Beleszynski and Thomas[36] claimed that if the concrete pore solution is rich in calcium ions, a calcium band forms around the reactive aggregate entrapping the alkali ions inside the reaction site (aggregate particle) and not allowing them out, consequently, causing expansion. The researchers found that in the presence of fly ash, calcium band is absent. A modification in gel chemistry, which leads to less expansive gel, was postulated.

Probably the most dominant mechanism is based on the entrapment of alkalis in the hydration products produced by pozzolans. The typical Ca-Si ratio of C-S-H in portland cements is approximately 1.8, however, reduced ratios are observed when pozzolan is introduced to system. The surface charge on C-S-H depends on its Ca-Si ratio: when the Ca-Si ratio is high, the surface charge of C-S-H is positive and anions are adsorbed on C-S-H (but not cations such as Na^+ and K^+, which remain in the pore solution available for ASR): when the Ca-Si ratio is lower than about 1.2–1.3, the surface charge of C-S-H becomes negative since alkali cations are incorporated on C-S-H in the presence of pozzolan.[35] As the removal alkali ions from the pore solution increases the extent of ASR occurrence decreases. Bhatty[41] postulated that in presence of pozzolanic materials, Ca-Si ratio is reduced by an increase in the amount of silica present in the C-S-H. Duchesne and Berube[42] also supported that as the Ca-Si ratio of the C-S-H decreases, more alkalis are removed from the pore solution and entrapped in the C-S-H gels. On the other hand, no consistent relationship is found between the C-S-H chemistry and the resulting ASR expansion.[39]

References

[1] Muller A (2004). Lightweight aggregates from masonry rubble. In: Conference on the Use of Recycled Materials in Building and Structures, Barcelona, Spain.

[2] Turley W (2002). C&D materials are recycled at an impressive rate (now if only America knew about it). C&D recycler. (www.findarticles.com)

[3] Muller A (2006). Recycling of construction and demolition waste—Status and new utilization methods. CODATA Beijing, China.

[4] Campbell JWP, Pryce W. Brick—A World History. London, UK: Thames Hudson, 2003.

[5] Plant Manager (2004). Glen-Gery Brick, Redfield, IA.

[6] Copernicus Research Project—CIPA-CT94-0211 (1998). Recycling of waste clay brick and tile material for the partial replacement of cement in concrete. Final Report.

[7] Mazumder AR, Kabir A, Yazdani N (2006). Performance of overburnt distorted bricks as aggregates in pavement works. Journal of Materials in Civil Engineering 18(6): 777–785.

[8] Toledo Filho RD, Goncalves JP, Americano BB, Fairbairn EMR (2007). Potential for use of crushed waste calcined-clay brick as a supplementary cementitious material in Brazil. Cement and Concrete Research 37(9): 1357–1365

[9] De Brito J, Pereira AS, Correia JR (2005). Mechanical behavior of non-structural concrete made with recycled aggregate. Cement and Concrete Composites 27(4): 429–433.

[10] Demir I, Orhan M (2003). Reuse of waste bricks in the production line. Building and Environment 38(12): 1451–1455.

[11] Schulz RR, Hendricks F (1992). Recycling of masonry rubble. In: TC Hansen (editor). Recycling of Demolished Concrete and Masonry. The International Union of Testing and Research Laboratories for Materials and Structures (RILEM) Report of Technical Committee 37-DRC Demolition and Reuse of Concrete. London, UK: Chapman & Hall.

[12] Poon CS, Chan D (2007). The use of recycled aggregate in concrete in Hong Kong. Resources Conservation & Recycling 50(3): 293–305.

[13] Khaloo AR (1994). Properties of concrete using crushed clinker brick as coarse aggregate. ACI Materials Journal 91(2): 401–407.

[14] Mansur MA, Wee TH, Cheran LS (1999). Crushed brick as coarse aggregate for concrete. ACI Materials Journal 96(4): 478–484.

[15] Khalaf FM, DeVenny AS (2004). Recycling of demolished masonry rubble as coarse aggregate in concrete: review. Journal of Materials in Civil Engineering 16(4): 331–340.

[16] Akhtaruzzaman AA, Hasnat A (1983). Properties of concrete using crushed brick aggregate. Cement and Concrete International 5(2): 58–63.

[17] Debieb F, Said Kenai S. The use of coarse and fine crushed bricks as aggregate in concrete. Construction and Building Materials, *in press*.

[18] Khatib JM (2005). Properties of concrete incorporating fine recycled aggregate. Cement and Concrete Research 35(4): 763–769.

[19] Padmini AK, Ramamurthy K, Mathews MS (2001). Behavior of concrete with low strength bricks as lightweight coarse aggregate. Magazine of Concrete Research 53(6): 367–375.

[20] Bektas F, Wang K, Ceylan H. Effects of crushed clay brick aggregate on mortar durability, Construction and Building Materials, *in review*.

[21] ACI Committee 232 (1994). Proposed Report: Use of natural pozzolans in concrete. ACI Materials Journal 91(4): 410–27.

[22] Baronio G, Binda L (1997). Study of pozzolanicity of some bricks and clays. Construction and Building Materials 11(1): 41–46.

[23] Sinton CW. Raw materials for glass and ceramics. Sources, process, and quality control. NJ: Wiley, 2006.

[24] Saad MNA, Andrade WP, Paulon WA (1982). Properties of mass concrete containing an active pozzolan made from clay. Concrete International 4(7):59–65.

[25] Ambroise J, Murat M, Pera J (1985). Hydration reaction and hardening of calcined clays and related minerals V. Extension of the research and general conclusions, Cement and Concrete Research 15(2): 261–268.

[26] Stein V. The raw materials for brick and tile making. In: W Beider, F Handle. Brick and tile making procedures and operating practice in the heavy clay industry: Chapter 6. Wiesbaden, Berlin: Bauverlag, 1982.

[27] Wild S, Khatib JM, O'Farrell M (1997). Sulphate resistance of mortar, containing ground brick clay calcined at different temperatures. Cement and Concrete Research 27(5): 697–709.

[28] Wild S, Gailius A, Hansen H, Pederson L, Szwabowski J (1997). Pozzolanic properties of a variety of European clay bricks. Building and Research Information 25 (3): 170–175.

[29] O'Farrell M, Wild S, Sabir BB (1999). Resistance to chemical attack of ground brick-PC mortar Part I. Sodium sulphate solution, Cement and Concrete Research 29(11): 1781–1790.

[30] O'Farrell M, Wild S, Sabir BB (2000). Resistance to chemical attack of ground brick-PC mortar Part I. Sodium sulphate solution, Cement and Concrete Research 30(5): 757–765.

[31] Turanli L, Bektas F, Monteiro PJM (2003). Use of ground clay brick as a pozzolanic material to reduce the alkali-silica reaction. Cement and Concrete Research 33(10): 1539–1542.

[33] Chen H, Soles JA, Malhotra VM (1993). Investigations of supplementary cementing materials for reducing alkali-aggregate reactions. Cement and Concrete Composites 15(1-2): 75–85.

[34] Xu GJZ, Watt DF, Hudec PP (1995). Effectiveness of mineral admixtures in reducing ASR expansion. Cement and Concrete Research 25(6): 1225–1236.

[35] Monteiro PJM, Wang K, Sposito C, Dos Santos MC, Andrate WP (1997). Influence of mineral admixtures on the alkali-aggregate reaction. Cement and Concrete Research 27 (12): 1899–1909.

[36] Beleszynski RF, Thomas MDA (1998). Microstructural Studies of Alkali-Silica Reaction in Fly Ash Concrete Immersed in Alkaline Solution. Advanced Cement Based Materials 7(2): 66–78.

[37] Wang H, Gillott JE (1991). Mechanism of alkali-silica reaction and the significance of calcium hydroxide. Cement and Concrete Research 21(4): 647–654.

[38] Ramachandran VS (1998). Alkali-aggregate expansion inhibiting admixtures. Cement and Concrete Composites 20(2-3): 149–161.

[39] Shehata MH, Thomas MDA, Beleszynski RF (1999). The effects of fly ash composition on the chemistry of pore solution in hydrated cement pastes. Cement and Concrete Research 29(12): 1915–1920.

[40] Ramlochan T, Thomas M, Gruber KA. (2000). The effect of metakaolin on alkali silica reaction in concrete, Cement and Concrete Research 30 (3): 339–344.

[41] Bhatty MSY (1985). Mechanism of pozzolanic reactions and control of alkali-aggregate expansion. Cement, Concrete, and Aggregates 7(2): 69–77.

[42] Duschesne J, Berube MA (1995). Effect of supplementary cementing materials on the composition of cement hydration products. Advanced Cement Based Materials 2(2): 43–52.

Chapter 3

Use of Ground Clay Brick as a Pozzolanic Material in Concrete

A paper to be submitted to Building and Environment (Elsevier)

F. Bektas[1], K. Wang[2], H. Ceylan[3]

Abstract

The studies to date showed ground clay brick (GCB) improves mortar durability, but the research on use of the GCB in concrete is still very limited. This study aimed to investigate the suitability of using GCB in concrete. Crushed clay brick originated from demolished masonry was ground in the laboratory and added to cement-based mixtures as partial cement replacement. Hydration characteristics of paste mixtures were investigated for a better understanding of concrete application. The effect of ground brick addition on concrete mechanical properties and chloride ion penetration resistance, as a measure of durability, were also studied. Two replacement levels, 15% and 25%, were compared with the control. The study on the cementitious pastes showed that the ground brick slightly increased the water demand for normal consistency, delayed the time of setting, reduced the temperature rise during hydration, and decreased the $Ca(OH)_2$ content. The tests on concrete showed that the mechanical properties (compressive, flexural and splitting tensile strengths and modulus of elasticity) of concrete containing ground brick were well comparable to those of the concrete without ground brick. Furthermore, the GCB increased the resistance of concrete to chloride ion penetration. The study

---

[1] Graduate student; primary researcher and author; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[2] Associate professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[3] Assistant professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

undertaken proved that, when it is finely ground, clay brick obtained from demolished masonry can be recycled as a pozzolanic cementitious material in concrete.

Keywords: Clay brick; Calcined clay; Supplementary cementitious material; Concrete

1. Introduction

Concrete is probably the most widely used construction material in the world. Billions of tons of natural resources (i.e., water, earth as aggregate and cement raw material) are consumed by the construction industry. Environmental considerations are pushing the industry toward more sustainable practices such as the use of recycled aggregate and industrial by-products, and the employment of less energy-intensive blended cements in concrete. Portland cements blended with pozzolanic materials (e.g., fly ash, silica fume, metakaolin, natural pozzolan) not only reduce clinker demand but also improve concrete durability such as resistance to sulfate attack and alkali-silica reaction.

Pozzolan is defined in ACI 116R as [1]:

> "a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperatures to form compounds possessing cementitious properties."

When used in portland cement systems, pozzolanic materials react with portlandite (or calcium hydroxide), which is a reaction product of portland cement and water, and forms secondary calcium-silicate-hydrate (C-S-H) gels, which in turn, improves internal structure such as refined porosity. Pozzolans are known to construction industry since the ancient Greeks. They used volcanic origin pozzolan-lime mixtures to build durable structures [1]. The predecessors of the Greeks, the Romans, refined the practice and discovered new pozzolan sources one of which is brick powder. When the natural pozzolanic material was not available the Romans used powdered brick to mix with lime and obtained a strong, durable binding material [2].

Some clays possess pozzolanic property when thermally treated: upon heating interlayer water is removed, crystal structure is destroyed, and a new material with pozzolanic property is formed. Particularly, kaolinitic clay demonstrates high pozzolanic activity: kaolin looses its structural water around 600 ^{o}C and metakaolin forms [3]. Calcined kaolin, or metakaolin, has been successfully used in mass concrete applications in Brazil since 1965. The production cost was one third of the portland cement and it suppressed the potential alkali-aggregate reaction in Jupia Dam [4]. Ambroise et al. stated that the most kaolin-rich and the mostly poorly crystallized clay shows the best pozzolanic activity [5]. Kaolin is an essential ingredient of clay brick manufacturing due to its sintering behavior [6]. Therefore, kaolin-rich clay brick may show pozzolanic activity in finely divided form and can be used as partial cement replacement material as a supplementary cementitious material.

Studies proved that finely ground clay brick improves the chemical resistance of mortar (e.g., sulfate attack and alkali-silica reaction) [7,8]. However, research including ground brick utilization in concrete applications is very limited. This study aims to demonstrate the suitability of using clay brick as a supplementary cementitious material in concrete. The origin of the brick used in the study was demolished masonry. It was ground in the laboratory and used in testing. Hydration characteristics of pastes containing various amounts of ground brick were investigated for a better understanding of concrete application. Mechanical properties and chloride ion penetration resistance of concrete mixtures with and without ground brick were determined in order to evaluate the effect of brick if used as partial cement replacement up to 25%.

2. Materials

A Type I portland cement was used to prepare paste and concrete specimens. Its specific gravity and Blaine fineness are 3.15, 368 m^2/kg, respectively. The chemical composition is given in Table 1. The clay brick tested as partial cement replacement was obtained in aggregate size. Its size was further reduced to passing 4.75-mm sieve (ASTM No. 4) and ground in a laboratory-type ball mill. A combination of steel balls with various diameters (12.5 mm, 25 mm and 40 mm) was used as the grinding medium. The chemical composition of the finely ground brick is given in Table 1. Its specific gravity is 2.68 and fineness is 370 m^2/kg (Blaine). 45-micron sieve (ASTM No. 325) residue has been measured as 10.2%.

Crushed oolitic limestone and natural siliceous aggregate were used to cast concrete specimens. The oven dry specific gravities are 2.45 and 2.59, and the moisture absorptions are 3.22% and 1.60% for coarse and fine aggregates, respectively. Fig. 1 shows the aggregate gradations—fine, coarse and combined.

3. Experimental Methods

The characteristics of ground clay brick (GCB) were investigated employing various techniques: fineness with Blaine apparatus (ASTM C 204—*Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus*), percent particles finer than 45-micron using wet sieving method (ASTM C 430—*Standard Practice for Fineness of Hydraulic Cement by the 45-micron (No. 325) sieve*), scanning electron microscopy (SEM) for visual inspection, chemical composition analysis with X-ray fluorescence (XRF), X-ray diffraction (XRD) for mineral composition.

Hydration characteristics of pastes (water + cementitious material) containing various amounts of GCB as cement replacement were determined. Three paste mixtures were prepared—0% (control: no GCB), 15% GCB (B15) and 25% GCB (B25). Water-to-cementitious ratio was 0.50. The pastes were mixed according to ASTM C 305—*Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency*. Normal consistency and setting times (initial and final) of the pastes were determined in accordance with ASTM C 187—*Standard Test Method for Normal Consistency of Hydraulic Cement* and ASTM C191—*Standard Test Method for Time of Setting of Hydraulic Cement by Vicat Needle*, respectively.

Semi-adiabatic temperature measurement was carried out in order to determine the effect of GCB addition on temperature rise during the hydration of cementitious pastes. Computer chips (or with the commercially known name, I-buttons) capable of measuring and storing temperature data were inserted into freshly mixed and molded pastes which were insulated by 50-mm thick styrofoam in order to minimize the heat interaction with the environment. The chips were set to collect data at every 3 minutes up to 60 hours.

The free calcium hydroxide, $Ca(OH)_2$, content of hardened cementitious pastes were determined by employing thermo gravimetric analysis (TGA) method. The pastes were cast into 25 × 50 mm cylindrical, capped PVC vials which were maintained at 23 ^oC until testing day—7, 28 and 91. At the testing day, the appropriate amount of material was extracted from the inner part and ground to pass 75-micron sieve (ASTM No. 200). TGA tests were performed employing a TA2950 Thermogravimetric Analyzer. The tests were carried out using approximately 50 mg of samples in dry nitrogen (N_2). A continuous heating with a rate of 10 ^oC/minute was applied to obtain the weight loss curve. A representative plot is given in Fig. 2. The relatively steep curve up to 100–110 ^oC corresponds to the loss of absorbed water. After that point non-evaporable structural water bound to C-S-H causes the gradual weight loss. The abrupt change in the curve roughly between 400 and 500 ^oC is due to the decomposition of $Ca(OH)_2$ which enables the quantification of the free $Ca(OH)_2$ in the paste.

At the next stage of the experimental study concrete mixtures were prepared to evaluate the effect of GCB on the mechanical properties. Similarly, 15% and 25% levels of GCB replacements (by weight of cement) were investigated. Similar to the pastes the concrete mixtures are labeled according to the GCB content—control, B15 and B25. Table 2 summarizes the mixture proportions. Compressive, flexural, splitting tensile strengths and modulus of elasticity of the concrete mixes were tested in accordance with ASTM C 39—*Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*, ASTM C 78—*Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)*, ASTM C 496—*Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens* and ASTM C 469—*Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression*, respectively. The tests were executed at 3, 7, 28, 91 and 180 days. 75 × 75 × 300 mm beams were used for the flexural testing and 75 × 150 mm cylinders were used for the others. In addition, ASTM C 1202—*Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration* was carried out to determine the effect of GCB addition on the chloride ion resistance of concrete. Tests were done using 100 × 50 mm disks aged to 28 and 91 days. All the specimens were stored in the moist room until the day of testing.

4. Results and Discussion

4.1. Characteristics of Ground Clay Brick

Due to its nature GCB can be defined as calcined natural pozzolan. According to ASTM C 618 a calcined natural pozzolan for use as a supplementary cementitious material, or mineral admixture as referred in the standard, in concrete has minimum 70% of silicon dioxide (SiO_2) plus aluminum oxide (Al_2O_3) plus iron oxide (Fe_2O_3), and maximum 4% of sulfur trioxide (SO_3), and maximum 10% of loss on ignition. The ground brick used in the study satisfies the chemical requirements of ASTM C 618: its total required oxide content, SO_3 content and loss on ignition are 92.06%, 0.04% and 0.16%, respectively.

Maximum 45-micron residue is limited to 34% according to ASTM C 618 and the mentioned value is 10.2% for the GCB. Blaine fineness of the GCB is identical to the Portland cement—370 m^2/kg. SEM imaging (Fig. 3) shows that the GCB particles as solid masses. Although the original material is porous in nature, grinding eliminates it. There are particles observed as big as 80 micron. Experience of the authors with the available laboratory ball mill shows that the brick would probably add no extra grinding energy cost if interground with portland cement clinker.

Fig. 4 gives the XRD profile of the GCB. High intensity quartz peaks around 21^o and 27^o are distinctive. There are also low-intensity hematite and albite peaks which can be associated to the presence of clay in the parent material. XRD profile implies the abundance of quartz. There are three essential ingredients of brick material—kaolinite, illite and quartz. Illite produces the plasticity while kaolinite acts as glue due to its good sintering property. It is possible to produce brick using only one of these clay types but usually a combination of different clay material is preferred. On the other hand, quartz is indispensable: it acts as stabilizer in the mix and limits drying shrinkage and improves void structure. In addition, fat clay rich bricks are known to be less frost resistant [6]. In the literature concerning brick XRD, cristobalite peak has been observed just next to the highest-intensity quartz peak [9]. The GCB used in the study did not show such peak. Kaolinite converts to metakaolin around 600 ^oC and to cristobalite when heated over 1000 ^oC [3]. Non-existence of cristobalite peak may be attributed to low firing temperature of the brick or a different clay chemistry or conversion of cristobalite over long time of periods [9]. The exact reason is unknown for the GCB used in this study and determination of it is beyond the scope of this study.

4.2. Normal Consistency and Time of Setting of Pastes

Table 3 gives the water-to-cementitious ratio required for normal consistency and associated time of setting of the cementitious pastes. The results showed that up to 25% GCB addition to paste does not cause significant water demand. Furthermore, as the GCB quantity increased the initial time of setting of the paste delayed. As for the final time of setting 15% GCB addition did not change the setting time compared to the control whereas 25% GCB increased the setting time. An increasing trend in the setting times was observed as the GCB is added to the pastes, however, the delays were not significant.

4.3. Temperature Measurement During Hydration

Cement hydration is an exothermic reaction that the temperature rise during the process may reach to significant levels. This concept is particularly important for mass concrete applications since temperature gradients through the mass concrete may be high enough to cause cracking. Fig. 5 shows the temperature measurements taken from the cementitious paste specimens. It is clear from the plot that the peak temperature decreases as the GCB amount in the paste increases. The peak temperatures recorded were 92.1 ^{o}C, 84.1 ^{o}C and 73.6 ^{o}C for the control, B15 and B25, respectively. The time at which the peak temperature attained was also modified by the GCB: 8.0hr, 9.0hr and 9.2hr for the control, B15 and B25 mixes, respectively. The delay in peak temperature implies that the GCB has a retarding effect on the hydration conforming the time of setting measurements.

4.4. $Ca(OH)_2$ Depletion in Pastes

Cement reacts with water to form C-S-H gel which is strong and stable acting as the glue of the concrete matrix. The reaction also produces calcium hydroxide $Ca(OH)_2$. $Ca(OH)_2$ is found in the form of octagonal crystals and generally rich in the aggregate-cement paste interfacial transition zone. These crystals are brittle and highly soluble, and therefore, they tend to reduce bond strength and durability.

Pozzolanic reaction consumes $Ca(OH)_2$ to produce secondary C-S-H gels. Therefore, $Ca(OH)_2$ measurement in pastes incorporating pozzolanic material provides information on pozzolanic activity. Fig. 6 shows the $Ca(OH)_2$ content of the pastes at the ages of 7, 28 and 91 days. At each day, the control sample has the highest amount of $Ca(OH)_2$ whereas the B25 sample has the lowest. It was found that the presence of GCB in the pastes decreases the $Ca(OH)_2$ considerably. For all the pastes, $Ca(OH)_2$ content increases from 7 days to 28 days implying the ongoing cement hydration, then, decreases to 91 days implying the finished or slow hydration. The increase rate from 7 to 28 days is higher for the control compare to those of the GCB pastes. Additionally, from 28 to 91 days is $Ca(OH)_2$ content of the control stays almost unchanged while there is significant decreases for the GCB pastes. This decrease indicates the depletion of $Ca(OH)_2$ through pozzolanic reaction in the presence of GCB.

4.5. Mechanical Properties of Concrete Mixtures

Tables 4–7 give the results of the mechanical testing of concrete mixtures—compressive strength, flexural strength, splitting tensile strength and modulus of elasticity. The data indicate that the GCB reduces early age strength, however, the strength gain rate is higher compared to the control that the ultimate strength values are closer or even higher than the control. For instance, at 3 days compressive strength ratios of B15 and B25 to the control are 0.87 and 0.78 which are in well agreement with the replacement ratios if it is assumed that only the portland cement hydrates. However, the same ratios are 0.99 and 0.94 at 91 days and 1.05 and 0.99 at 180 days for B15 and B25, respectively. Similarly, the mentioned ratios are higher at the later ages compared to the early ages for the flexural and splitting tensile strengths. This behavior is well expected since the pozzolanic materials (or supplementary cementitious material) produces secondary C-S-H gels for strength development. As for the static modulus of elasticity of the mixtures the GCB incorporated mixtures show slightly lower values compared to no GCB mixture: 1–10% varying with the age and GCB content. At 180 days, B15 and B25 attained 97% and 94% of the control's modulus of elasticity, respectively. The testing showed that up to 25% GCB addition produces comparable strength values to those of the control.

4.6. Resistance of Concrete Mixtures to Chloride Ion Penetration

ASTM C 1202 results are given in Fig. 7. The 91-day mixtures achieved higher resistance to chloride penetration than the younger 28-day ones: the trend was true for all the mixtures tested. The improvement can be explained on the basis of continuing hydration. The plot also clearly demonstrates the positive effect of GCB addition on the chloride ion resistance of concrete. As the GCB content increases the ion passed through concrete decreases. In other words, the B25 mixture, which has the highest amount of ground brick, showed the best performance. In addition, from 28 to 91 days, B25 mixture demonstrated the highest improvement which may be attributed to denser internal structure caused by the pozzolanic C-S-H gels.

The concrete testing showed that it is well possible to produce concrete mixtures with adequate mechanical properties employing up to 25% of powdered clay brick obtained from demolished masonry. In addition, GCB considerably reduces the chloride ion penetration of concrete which most possibly indicates it would improve concrete durability. Additional environmental benefits can be expected with the use of GCB in concrete mixtures: the benefit is two-fold—reduction in portland cement clinker demand and removal of demolished masonry wisely.

5. Conclusion

Based on the experimental study investigating the use of GCB in concrete, the following conclusions, which are limited to the materials used in the study, were drawn:

- Up to 25% GCB replacement for cement has no significant effect on the water demand for normal consistency and on the time of setting.
- GCB replacement reduces the heat dissipated during cementitious hydration.
- $Ca(OH)_2$ content decreases as GCB amount increases in the pastes, which is partially attribute to the pozzolanic action between the GCB and cement.
- Similar to concrete with other pozzolanic materials, concrete with GCB had a little low compressive strength at early age but comparable strength to the

concrete with no GCB at late age. Other mechanical properties of the GCB concrete are also comparable to those of control concrete with no GCB.

- GCB improves the resistance of concrete to chloride ion penetration. The improvement is proportional to the GCB quantity.
- Clay brick originated from demolished masonry can be recycled as a pozzolanic material for cement replacement in concrete.

References

[1] ACI Committee 232. Proposed Report: Use of natural pozzolans in concrete. ACI Materials Journal 1994; 91(4):410–27.

[2] Baronio G, Binda L. Study of pozzolanicity of some bricks and clays. Construction and Building Materials 1997; 11(1):41–6.

[3] Sinton CW. Raw materials for glass and ceramics. Sources, process, and quality control. NJ: Wiley; 2006.

[4] Saad MNA, Andrade WP, Paulon WA. Properties of mass concrete containing an active pozzolan made from clay. Concrete International 1982; 4(7):59–65.

[5] Ambroise J, Murat M, Pera J. Hydration reaction and hardening of calcined clays and related minerals V. Extension of the research and general conclusions, Cement and Concrete Reearch 1985; 15(2):261–8.

[6] Stein V. The raw materials for brick and tile making. In: W Beider, F Handle. Brick and tile making procedures and operating practice in the heavy clay industry: Chapter 6. Wiesbaden, Berlin: Bauverlag; 1982.

[7] O'Farrell M, Wild S, Sabir BB. Resistance to chemical attack of ground brick-PC mortar: Part I. Sodium sulphate solution. Cement Concrete Research 1999; 29(11): 1781–90.

[8] Bektas F, Turanli L, Wang K, Ceylan H. Comparative performance of ground clay brick in mitigation of alkali-silica reaction. ASCE Journal of Materials in Civil Engineering, in press.

[9] Livingston RA, Stutzman PE, Schumann. Quantitative x-ray diffraction analysis of handmolded brick. In: NS Baer, S Fritz, RA Livingston, editors. Conservation of Historic Brick Structures: Chapter 11. Shafesbury, UK: Donhead Publishing; 1998.

[10] Ramlochan T, Thomas M, Gruber KA. The effect of metakaolin on alkali-silica reaction in concrete. Cement Concrete Research 2000; 30(3): 339–344.

List of Figures

List of Tables

Table 1. Chemical composition of cementitious materials

Composition (%)	Portland Cement	Ground Clay Brick
CaO	64.61	0.81
SiO_2	20.58	69.90
Al_2O_3	5.38	15.38
Fe_2O_3	2.14	6.78
MgO	2.08	1.58
SO_3	3.01	0.04
K_2O	0.46	2.78
Na_2O	0.26	1.02
Loss on ignition	1.00	0.16

Table 2. Mixture proportions of concrete mixes

Mix Label	Control	B15	B25
Portland cement (kg/m^3)	400	340	300
Ground clay brick (kg/m^3)	-	60	100
Water (kg/m^3)	180	180	180
w/cm	0.45	0.45	0.45
Combined aggregate (kg/m^3)	1750	1750	1750

Note: w/cm=water-to-cementitious ratio

Table 3. Water-to-cementitious ratio for normal consistency and time of setting

	w/cm for normal consistency	Time of setting (min)	
		Initial	Final
Control (0% GCB)	0.275	135	190
B15 (15% GCB)	0.281	140	190
B25 (25% GCB)	0.286	145	195

Table 4. Compressive strength of concrete mixtures

	Control	B15	B25
3 days	28.0	24.3	21.9
7 days	37.9	32.2	30.5
28 days	42.8	41.1	38.4
91 days	48.3	47.9	45.3
180 days	47.0	49.2	46.4

Note: All values are in MPa.

Table 5. Flexural strength of concrete mixtures

	Control	B15	B25
7 days	5.1	4.4	4.4
28 days	5.1	4.8	5.2
91 days	5.4	5.3	5.2
180 days	5.8	5.7	5.5

Note: All values are in MPa.

Table 6. Splitting tensile strength of concrete mixtures

	Control	B15	B25
7 days	3.2	2.9	2.4
28 days	3.5	3.8	2.7
91 days	4.0	4.2	3.7
180 days	3.9	4.1	3.8

Note: All values are in MPa.

Table 7. Modulus of elasticity of concrete mixtures

	Control	B15	B25
3 days	26.1	25.8	24.0
7 days	29.5	27.3	26.5
28 days	30.9	30.0	28.7
91 days	32.9	31.7	31.6
180 days	34.0	33.1	32.0

Note: All values are in GPa.

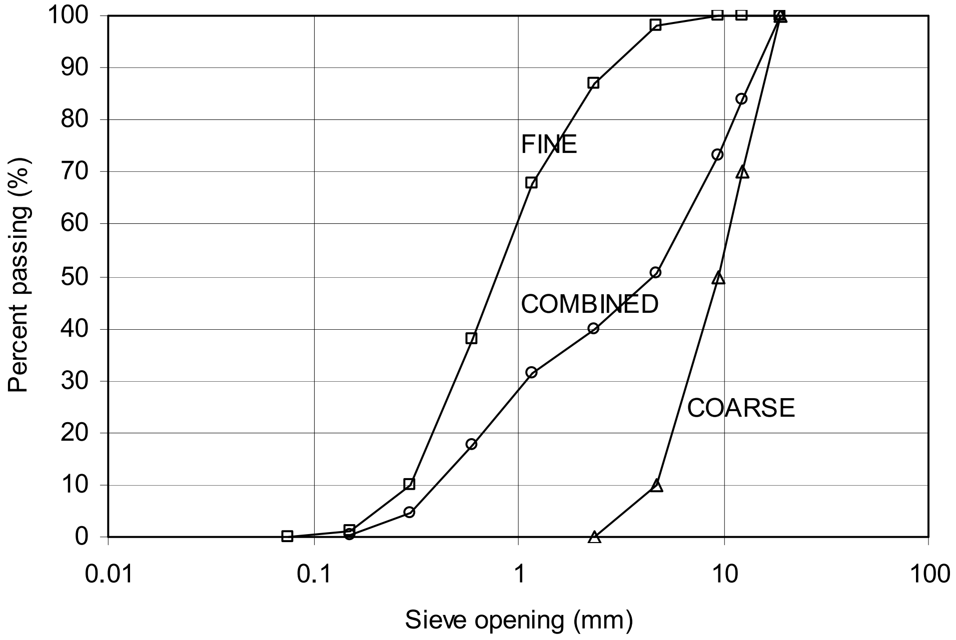


Fig. 1. Aggregate gradations used in the concrete mixtures.

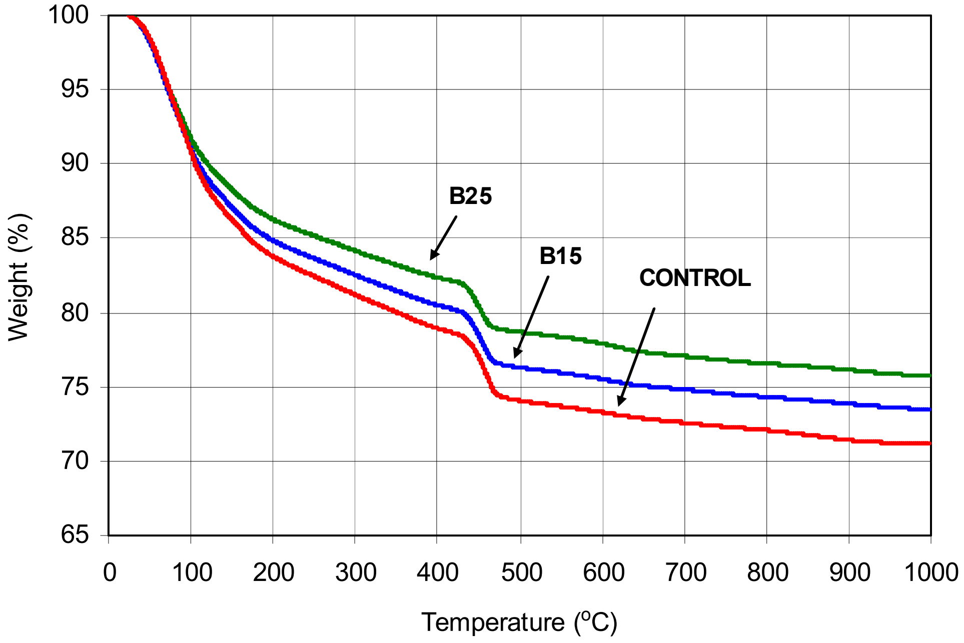


Fig. 2. TGA output of the paste samples tested at 91 days.

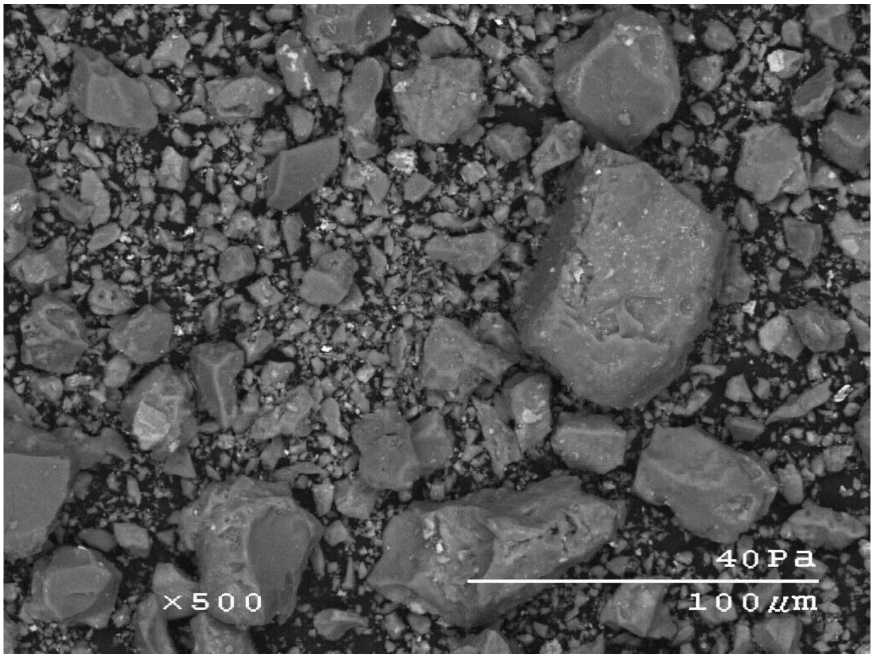


Fig. 3. SEM image of the ground clay brick.

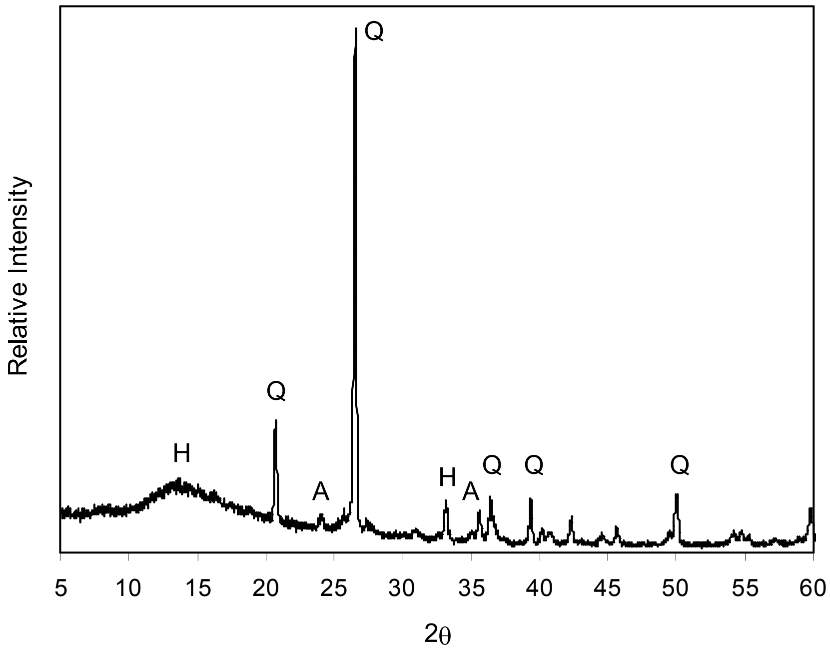


Fig. 4. XRD profile of the ground clay brick (Q=quartz; H=Hematite; A=Albite).

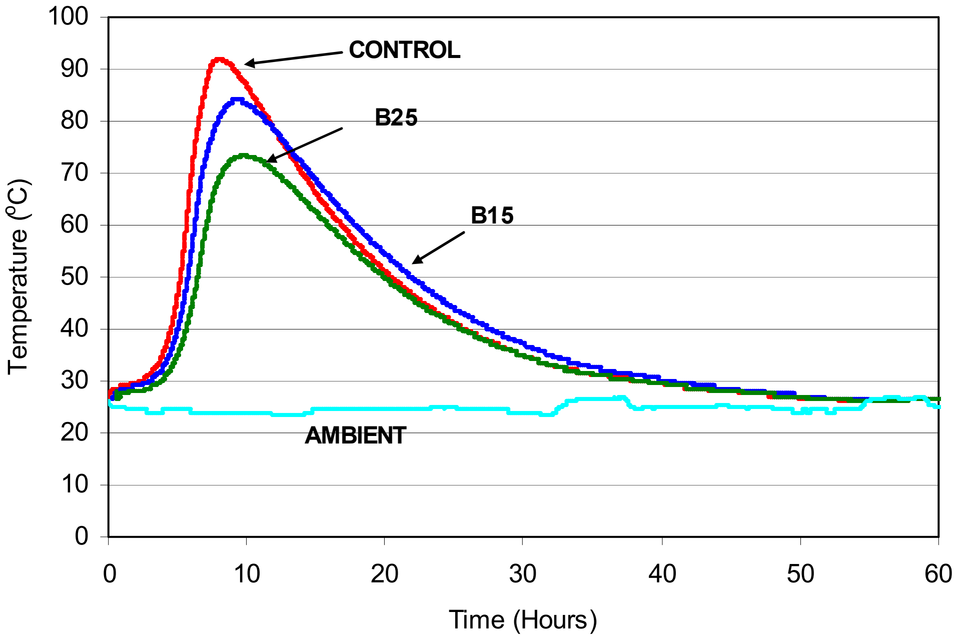


Fig. 5. Temperature rise during the hydration of the cementitious pastes.

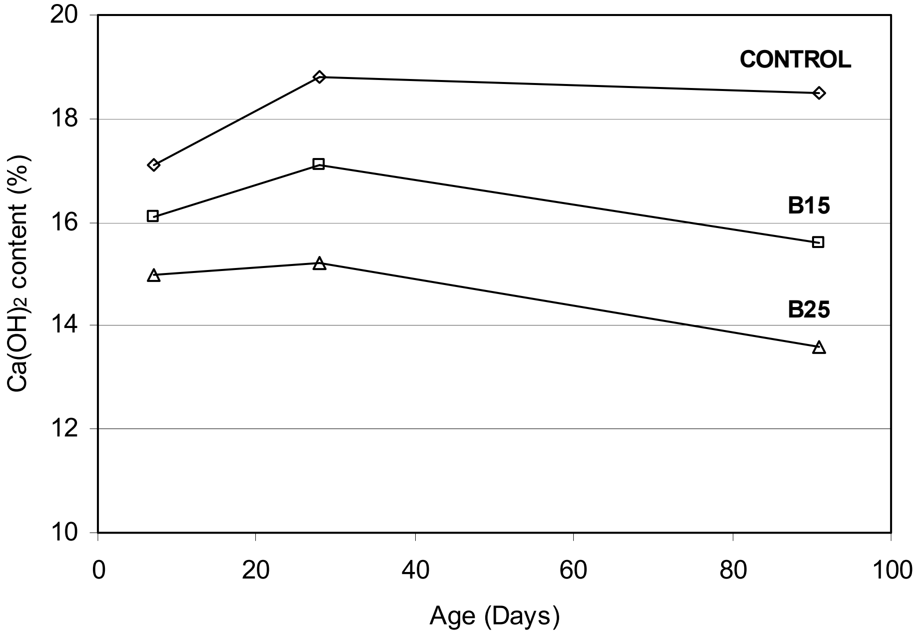


Fig. 6. $Ca(OH)_2$ content calculated from TGA results.

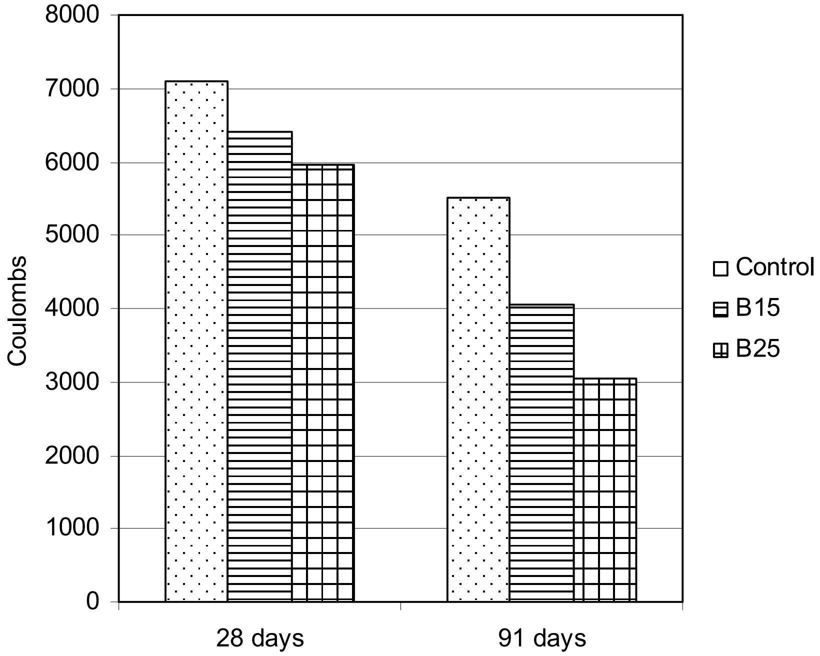


Fig. 7. Chloride ion penetration (ASTM C1202) of the concrete mixtures.

Chapter 4

Comparative performance of ground clay brick in mitigation of the alkali-silica reaction

A paper accepted for publication in Journal of Materials in Civil Engineering (ASTM)

F. Bektas[1], L. Turanli[2], K. Wang[3], H. Ceylan[4]

Abstract

This paper reports on a study of waste clay brick that was ground and used as a supplementary cementitious material (SCM) in a mortar. The effect of this ground brick, in comparison with other conventional pozzolanic materials (namely, a Class F fly ash, and a natural pozzolan), on the alkali-silica reaction (ASR) of the mortar was evaluated. The ground clay brick, fly ash, and natural pozzolan were introduced into the mortar as a cement replacement at a 20 or 35% level. Fly ash - natural pozzolan and fly ash-ground clay brick combinations were also employed as a cement replacement at a 35% level. These SCM-incorporated mortar samples were tested for ASR expansion, flexural and compressive strength, and rapid chloride permeability. The test results indicate that all the SCMs considered in this study reduced the mortar ASR expansion, rapid chloride permeability, and the ASR-associated strength loss. The finely ground waste clay brick demonstrated high potential to reduce the ASR expansion as well as the ASR-associated strength loss.

CE Database subject headings: Cements; Clays; Bricks; Mortars.

---

[1] Graduate student; primary researcher and author; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.
[2] Associate professor; Department of Civil Engineering, Middle East Technical University, Ankara, Turkey 06531.
[3] Associate professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.
[4] Assistant professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

Introduction

The cement industry releases 1.6 billion tons of cement to the market annually. One ton of portland cement production corresponds to one ton of carbon dioxide (CO_2) emission. As a result, the cement industry produces about 5% of global, man-made CO_2. Half of the CO_2 is from the chemical process of clinker production, 40% from burning fuel, and the remaining 10% is split between electricity use and transportation (WBSCD 2005). Minimization of the portland cement clinker production would contribute a significant reduction in the global CO_2 emission.

Use of supplementary cementitious materials (SCMs), either as blending agent in cement production or as cement replacement in concrete production, helps to reduce clinker consumption significantly. Certain cementitious materials such as fly ash or slag can be used up to 70% mass of cement. Use of these materials in concrete production not only decreases the demand for portland cement clinker but also eliminates the problem of waste disposal. Furthermore, it is well known that these pozzolanic materials provide technical benefits to concrete (e.g., increased strength and, most importantly, enhanced durability.).

Waste clay brick is one of the potential sources of pozzolanic cement replacement. In addition to those obtained from the demolition of existing buildings, the brick/tile manufacturing process also produces a significant amount of waste brick. Some portion of the fired material is discarded due to being offstandard—broken, underburned, or overburned, and mostly dumped around the brick factories (Khaloo 1994). In the last decade, there has been a growing revisit to the use of waste brick or more generic name ceramic (Chen et al 2003; De Brito et al 2005; Poon et al. 2006; Khalaf and DeVenny 2005; Khatib 2005). These studies have particularly focused on using discarded brick as concrete aggregate. There are also a few utilizations of the waste brick as a pozzolan source. Siliceous and aluminous clays possess pozzolanic property when thermally treated (such as metakaolin). During a heat treatment, the crystal structure of the clay minerals is destroyed, and an amorphous or disordered alumino silicate structure is formed leading to pozzolanicity (Mehta 1984). The clay brick manufacturing process is simply a pozzolan production. When the brick is ground to adequate fineness it can be used as a SCM for concrete. This concept is dated back to the ancient Romans. They used lime mortars containing crushed brick and brick powder in floors and foundations for water proofing purposes (Baronio and Binda 1997). The Romans called this mortar as cocciopesto which was inherited as horasan by the Ottomans. Surkhi and homra are the names used in India and in the Arabian Peninsula, respectively. The use of brick in mortar continued in the Byzantine structures preceding the Ottomans'. Recent studies have proved that the crushed brick-lime mortar also serves for structural purposes particularly for earthquake resistance due to improved ductility (Moropoulou et al. 2002).

The ground clay brick has been the concern of several studies aimed at utilizing it in modern concrete. The ground clay brick was found to improve the micro-structure of the mortar leading to an increase in compressive strength. The strength improvement was achieved by the refinement of the pore structure and/or pozzolanic action (O'Farrel et al. 2001; Heikal 2000). Furthermore, O'Farrel et al. (1999; 2000) investigated the ground clay brick to enhance mortar resistance against chemical attacks of sulfate and seawater. Prior to the present study, the first two writers of this paper, together with Monteiro, utilized the ground clay brick to mitigate deleterious alkali-silica reaction (ASR) and found a consistent reduction in the expansion as the brick quantity increases in the binder mass (Turanli et al. 2003). In another subsequent study, they interground up to 35% (by mass) clay brick with portland cement clinker to produce blended cements and obtained promising results in mortar strengths and ASR mitigation (Bektas et al. 2004). To date, the benefits of ground clay brick against ASR have been investigated as its effect on the expansion, however, the ASR-affected mechanical properties have unlikely been the focus.

The present study is a continuation of the previous efforts to improve concrete durability using ground waste clay brick. An experimental study was carried out to compare the performance of ground brick with that of fly ash and natural pozzolan in terms of ASR resistance. The ground clay brick was tested for ASR resistance in binary and ternary binder mixes in comparison with the other SCMs. Expansion measurements were done up to 12 months on the samples stored in sodium hydroxide solution at 80^{o}C. In addition, the effect of SCMs on the mortar strength in the presence of the deleterious ASR was also evaluated.

Materials

The ordinary portland cement (OPC) used corresponds to CEM I 42.5 N and Type I according to EN 197-1 and ASTM C150, respectively. Class F fly ash (FA), natural pozzolan (NP), and ground clay brick (GCB) were the SCMs utilized in combinations with the OPC. The chemical composition of the fine materials is given in Table 1. Low-calcium FA was obtained from a national thermal power plant in the north-western region of Turkey and used as it was delivered. NP used in the study was produced from volcanic tuff whose pozzolanic activity has been proven in a previous research (Uzal and Turanli 2003). Crushed clay brick which is being sold in the market for various applications (e.g., landscaping, running track surfacing) was the parent for GCB. The production processes of the NP and the GCB were identical: the raw materials, volcanic tuff and crushed brick, were reduced in size using a jaw crusher. They were then sieved and the material retained on No.12 sieve (1.70 mm) was discarded in order to control the maximum particle size. A batch of 10 kg brick was ground in a laboratory type ball mill. The grinding media were steel balls and cylinders. The fineness of the FA, 27±3% residue on 45 µm (ASTM No.325) sieve, was taken as reference for the NP and the GCB. The Blaine finenesses of these materials were also measured, and they are 310 kg/m^2 (with a specific gravity of

3.03), 340 kg/m^2 (2.25), 480 kg/m^2 (2.26), and 290 kg/m^2 (2.64) for OPC, FA, NP, and GCB, respectively.

In the experimental program, perlite and limestone were employed as the mortar aggregate. Limestone is composed of 100% calcite ($CaCO_3$) and totally alkali inert. The perlite sample studied is white in color, glassy with choncoidal fracturing, and includes pumice and granite rock fragments together with dark mica phenocrysts. Under the microscope, the sample displays poorly developed flow and perlitic texture (Fig.1). The aggregates were sieved according to ASTM C1260 and blended according to the pessimum content of the perlite to obtain alkali-reactive mortar.

Experimental Program

Mix Proportions

To establish the effectiveness of GCB durability as a SCM, an experimental program was designed to compare it with FA and NP. FA has been chosen since it is the most well known and commonly used pozzolan in the construction industry throughout the world. NP has been included in the program since it is locally abundant and the most widely used blending agent in Turkish cement industry. There was a total of nine binder mixes tested: one is the control mix, containing only OPC; six are binary mixes, in which OPC was individually replaced by FA, NP, and GCB at the replacement levels of 20% and 35% (by mass); and the other two are ternary mixes of 65%OPC + 20%FA + 15%NP and 65%OPC + 20%FA + 15%GCB. The idea behind the use of FA in combination with another SCM is that the high quality FA (low-calcium content Class F) is scarce in Turkey and to obtain low-calcium FA is costly in most construction projects. Therefore, a certain part of the FA in the blend was replaced with either GCB or NP in order to evaluate the behavior of such a ternary binder blend.

The aggregate was sieved and recombined according to ASTM C1260 gradation. The same mortar mix proportion was utilized in the preparation of all the tests—ASR expansion, strength and chloride permeability. Cementitious-to-sand ratio (cm/*s*) was 1:2.25 and water-to-cementitious ratio (*w*/cm) was 0.47 as recommended by ASTM C1260.

Tests

The experimental program basically included two types of measurements: expansion and strength. The expansion measurements were carried out in accordance with the accelerated mortar-bar method, ASTM C1260. The mortar bars were cast and demolded after being stored at room conditions for 24 h. They were put into a water bath whose temperature was gradually raised to 80±2°C and cured for another 24 hours. The bars were then immersed in 1 N NaOH solution at the same temperature and the expansions were recorded up to 12 months.

Polished samples were prepared from the control bar and studied under scanning electron microscope (SEM) equipped with energy dispersive X-ray spectrometer (EDS). Basic information on the alkali reactivity of the perlite aggregate was obtained. However, detailed investigation of the cementitious material-perlite reaction is beyond the scope of this study.

The mortar flexural and compressive strengths were determined in accordance with ASTM C348 and ASTM C109, using 40 × 40 ×160 mm mortar prisms and 50 mm cubes, respectively. The samples were stored in either lime saturated water at 23°C or 1 N NaOH solution at 80°C until testing. For the samples stored in NaOH solution, 1 mortar volume to 4±0.5 solution volume was utilized as recommended for the storage of mortar bars in ASTM C1260. The samples were tested for compressive strength at 7, 28, and 91 days after casting.

Rapid chloride permeability test, in accordance with ASTM C1202 procedure, was done on the mortar mixes containing 35% cement replacements. Mortar disks of 50 mm in thickness and 100 mm in diameter were cast for this test. Two curing conditions were applied: 28 days at 23±2^{o}C and 7 days at 80±2°C. The samples were brought to room temperature before testing.

Results and Discussions

Alkali Reactivity of Perlite

Perlite is a volcanic siliceous rock formed by the rapid cooling of lava or magma which causes concentric spherical cracks that give rise to an onion-skin structure. Its high water content, 3-5%, distinguishes perlite from the other volcanic glasses such as obsidian, pumicite etc. Its volcanic origin together with the deformed crystal structure makes perlite potentially alkali-reactive when used as concrete aggregate.

Turkey has large perlite deposits and is one of the most important perlite producers in the world. Perlite is vitrified to produce a lightweight material called expanded perlite which is the well-known form in the construction industry and used as lightweight aggregate or as a plaster ingredient for insulating purposes. On the other hand, in Turkey, some applications of the perlite rock as concrete aggregate are known to the knowledge of the writers, however, no ASR case has been reported. Suitability of the perlite as concrete aggregate is also beyond the scope of this study.

A series of mortar bars, in which the perlite aggregate was replaced by limestone aggregate in quantities varying from 1% to 100%, was subjected to ASR testing in order to evaluate the reactivity of the perlite. Expansion versus perlite content is plotted in Fig.

2 for different ages. Like other water bearing, highly siliceous minerals such as opal and obsidian, perlite was found to be alkali reactive with pessimum content. The expansion proportionally increases with the reactive material content and tends to decrease after a peak point which is the pessimum amount, around 30% in this case, as seen in Fig. 2. Therefore, the blended aggregate, 20% perlite and 80% limestone (or perlite:limestone = 1:4 by weight), was utilized in the present study for the evaluation of the SCMs. After 12 months of exposure, the control mortar bars, with no SCM, were severely damaged by ASR and the cracks could be detected visually: the openings were up to 1 mm. Microstructural investigation clearly demonstrated the presence of reaction and associated cracking. Fig. 3 gives a general picture of the microstructure. The reacted aggregate particles were connected with gel-filled cracks. It is apparent that the gel was flowable so that the voids were either lined or completely filled with this reaction product. Although there were a few limestone particles affected by the cracking initiated from the perlite aggregates, surprisingly the limestone particles were mostly intact.

Fig. 4 shows a reacted particle turned into a gel; there is light-colored rim around the aggregate. The EDS analyses have shown that this band is very rich in calcium (Fig. 4—spectrum 1). On the other hand, the inner gel is rich in alkali, both sodium and potassium, and has very little or no calcium (Fig. 4—spectrum 2). Moreover, the inner reaction site contains much more potassium compared to the boundary. The aggregate cement interface is the place where calcium hydroxide is abundantly found. Perhaps, the ion exchange occurs between the cement paste and the reaction gel, with the gel becoming an alkali-calcium-silica complex at the aggregate boundary. Bleszynski and Thomas (1998) stated that the high calcium gel found at the periphery of reactive aggregate forms a semi-permeable membrane which permits the diffusion of alkali ions into the particle but prevents the counter-diffusion of resulting gel. Thus, an expansive force is created inside the rim by the gel.

ASR Testing with SCMs

As mentioned previously, limestone and perlite aggregates were blended at a ratio of 1:4 by mass in order to obtain the ASR expansive mix to test the efficacy of the SCMs (FA, NP, and GCB) which were introduced as cement mass replacement up to 35%. The ASTM C1260 procedure was followed for the ASR testing. In fact, a new standard, ASTM C1567—*Standard test method for determining the potential alkali-silica reactivity of combinations of cementitious materials and aggregate (accelerated mortar-bar method)* has identical procedure to the one used in the study. However, the standard had not been released when the experimental program started.

The ASR expansion curves of the mortar bars tested with various combinations of the mineral admixtures are given in Figs. 5-7. In each figure, plot (a) contains measurements of the first 56-days and (b) has the data up to 12 months. Figs. 5-8 include the data for the individual 20 and 35% replacements of FA, NP, and GCB, respectively. Fig. 8 contains the curves for 20%FA+15%NP and 20%FA+15%GCB additions. The expansion curve of the control mix, containing no SCM, is also given in each plot.

ASTM C1567 classifies the mixes having an expansion below 0.10% at 14 days as not being deleterious, hence, indicating the mitigation measure as effective. In addition, ASTM C1260 classifies the aggregates as innocuous expanding below this limit. Therefore, it would not be misleading to use the 0.10% limit in the analysis of the results of this study. According to this criterion, 20% replacement of all the replacement materials has failed the test although expansion reductions have been achieved. It is worth noting that, at the 20% level, FA was very close to 0.10%, being 0.12%, whereas the others were approximately five-times the limit. On the other hand, both individual and combined replacements of FA, NP, and GCB at 35% have shown superior performance suppressing the deleterious reaction, meaning the expansions were restrained below 0.10% at 14 days. Based on the fuel material, the FA used in the study was the only available Class F found in Turkey (Turker et al. 2004). High-quality FA for concrete applications is relatively scarce in Turkey, therefore, blends of FA and NP, or GCB were tested for ASR to evaluate the potential for future applications. The results showed that the ternary blends of OPC (65%), FA (20%), and NP or GCB (15%) worked well. The expansions for both combinations were approximately 3% of the control.

The expansion of the control sample almost leveled off at 56 days. At this age, the expansion experienced by the 20% replacement of all the admixtures was very close to being 0.87, 0.84, and 0.93% for FA, NP, and GCB, respectively. This picture is very different than the one at 14 days. Fig.5a clearly demonstrates that the mortar with 20% FA experienced a steep expansion curve after 14 days reaching to the same level as 20% NP and GCB at 56 days. Figs. 5(b), 6(b), 7(b) contain the data up to 1 year. For the mortar bar containing 35% SCM replacements, it is seen that the reaction and the associated expansion continued after 56 days. Numerically the expansion values were still lower compared to the control sample; however, a comparison between expansion values might be misleading at this point since the bars were severely crooked after 1 year of exposure and the linear expansion might not be representative at this stage. Nonetheless, with the prolonged exposure to NaOH solution the reaction progresses in the bars containing 35% additives. Barringer (1999) also questioned whether the 14 day test period gives a good indication of the potential for an aggregate to react; however it leaves question as to the effect of preventive measures to suppress this reaction. Similarly, Berube et al. (1995) concluded that the accelerated mortar bar method is viable to test SCMs as long as the testing period is kept at 14 days and the reduction in pore solution alkali provided by the SCMs still holds. SCMs probably reduce the alkalinity of the pore solution initially, and also decrease the permeability limiting the alkali ion penetration from the immersion NaOH solution. However, once the cracking begins the ion mobility also starts to increase. Then, the reaction happens to take place at the inner regions and the SCM no longer be beneficial with the increased cycle. The permeability concept will be revisited in the coming section of this paper. In this study, the prolonged NaOH exposure has proved that the pozzolanic additives may not be helpful in mitigating the reaction when there is a source of continuous alkali supply. Deicing materials based on alkali compounds might be a good example. For instance, NaCl, which is used as deicing agent on pavements, is well known to exacerbate the ASR (Katayama et al. 2004; Berube et al. 2003). In addition, new generation potassium- and sodium-acetate based deicing chemicals are extensively used on airfield runways. These solutions have been proved to increase the rate of deleterious ASR (Rangaraju et al. 2005). Deicing chemicals are continuously applied in the wintertime which results in the saturation of concrete with alkalis. In such a case, ASR may take place sooner or later no matter how high the percentage of pozzolanic additives is utilized if the aggregate is potentially reactive.

*Strength Test*s

The flexural strengths of the mortar prisms stored either in water at room conditions or in 1 N NaOH solution at 80°C are given in Table 2. When compared to the control mortar, at the early age, the mortars containing 20% SCMs have higher values, whereas the ones with 35% replacement have lower values except for GCB: 35% GCB did not reduce early age flexural strength. For all the mixes, the strength was improved with age. At the end of 91 days, the values of the admixture mixes were almost equal to or higher than that of the control mix. The ternary blends also achieved the same ultimate strength as that of the control.

The beneficial effect of the SCMs on the mechanical properties in the presence of deleterious ASR could be clearly observed beginning from an early age. All the mortar samples with SCMs had higher flexural strength than that of the control at up to 91 days. When the results of water cured and solution cured samples at 7 days are compared, it is seen that the control mix experienced a strength loss due to the ASR, whereas other mixes possessed significant strength gains. The SCMs were effective against ASR preventing severe cracking, and the elevated temperature also promoted the pozzolanicity: the values were even higher than those of the water cured for 91 days. In addition, an alkali environment might also promote the pozzolanic activity: alkalis are known to be good activators for the pozzolanic materials like fly ash, slag, and natural pozzolan. GCB was not temperature sensitive as FA and NP were. As the reaction progresses with time, thus cracking becomes severe; the strength reduces at later ages. At 91 days the cracking was effective for all the mixes, probably controlling the permeability of the mortars. However, the values of the SCM samples were still dramatically higher, even 2-3 times than that of the control mortar. At this point, the writers would like to mention that the variation in the testing results increased at the later ages. Therefore, six samples were averaged for the ASR curing in order to control the fluctuation in the results, and even the tests were repeated in certain cases. Twenty eight days can be considered as the optimum age to show the benefits of the cement replacements used: at this age, the expansion of the control bars was significantly high being 1.21%, however, the SCMs were still very effective and not dominated by the excessively harsh solution.

The results of the compressive strength tests obtained from 50 mm cubes are summarized in Table 3. Unlike the flexural strengths the compressive strength of the control sample showed no reduction at 7 days. On the other hand, similar to the flexural capacity, compressive strength of the SCM samples stored in the NaOH solution demonstrated higher values compared to the water cured samples, probably due to the accelerated pozzolanic activity provided by the hot solution. At later ages, the trend was not clear as in the case of flexural strength. Although the cracking was visible reaching 1 mm after 91-day exposure of a severe NaOH solution the compressive strength results did not reflect this observation. This finding is also confirmed by Jones and Clark stating that the cube test is insensitive to ASR expansion (Jones and Clark 1998). Ahmed et al. (2003) also concluded that the compressive strength is not a good indicator of ASR, whereas the tensile strength and the elastic modulus are very sensitive to the deterioration.

Table 4 is more helpful to visualize the beneficial effect of SCMs on the mechanical strength loss due to ASR. The value indicates the ratio of the ASR cured sample to the corresponding water cured one at the same age. The values greater than 1.0 correspond to strength improvement and less than 1.0 strength show reduction due to deleterious ASR. At 7 days the SCM mixes showed strength increase, whereas the control samples experienced loss, and this is valid for both flexural and compressive strengths. At 28 days, loss in flexural strength is 67% for the control sample. This value is only 7, 26, 10, 6, and 10% for the 35% replacements of FA, NP, GCB, FA+GCB, and FA+NP, respectively. The performance of SCM mixes is also better compared to the control mixes at 91 days. As for the compressive strength, the strength reduction is 23% at 28 days. On the other hand, 35% SCM added mortars showed strength development. At 91 days, the strength loss in the control sample was 23% and for SCM mixes the value was in the range of 12-32%.

The results demonstrated that all the cement replacement materials (i.e., FA, NP, and GCB) help to reduce the negative effects of ASR on the mechanical properties. It has been found that SCM addition is much more helpful to maintain flexural strength than it is for compressive strength. The performance of FA is the best to suppress ASR; on the other hand, GCB outperforms or is comparable to NP in the tests.

Rapid Chloride Permeability Test

ASTM C1202 procedure was followed to test the chloride permeability of the mixes. The tests were performed on the samples either cured at 23^{o}C for 28 days or at 80^{o}C for 7 days. The impetus was to evaluate the 80^{o}C temperature effect on the permeability of the mixtures, if any. The strength increase experienced at 7-day NaOH curing can be attributed to this temperature curing. The results are given in Table 5. For both of the curing conditions, the mortars having SCMs performed better compared to the control mix. Dramatic reductions have been achieved, particularly at high temperature curing. The results are in good agreement with the ASR performance: FA performed the best, with NP and GCB being similar, however, superior to the control. The temperature effect on the cement replacements was significant: high-temperature curing resulted in reduced ion penetration. This improvement is attributed to the increased pozzolanic activity. Previous research has confirmed that the elevated temperature curing promotes the pozzolanic activity and thus reduced permeability (Thomas et al. 1989). Obviously, reduced permeability helps the mortar to resist ASR, inhibiting the ion mobility. The effect of elevated temperature on GCB was lower compared to the others. Moreover, the performance of OPC is negatively affected by increased temperature: the values were so high that the measurements had been repeated; even some could not be measured, probably, due to high permeability. Microcracking due to thermal stresses as a result of high temperature might be the reason for this anomaly (Balenderan and Martin-Buades 2000). It is worth to note that the electrical conductivity or resistivity of the concrete measured by the ASTM C1202 cannot be directly related to the permeability of the material. For this the procedure is criticized, particularly, in the presence of mineral admixtures (Feldman et al. 1999; Shi 2004; Julio-Betancourt and Hooton 2004). However, the results can be partially related to the permeability but not completely. In addition, the standard test has been widely accepted and still used in research for comparison purposes.

Summary

This study aimed at evaluating the potential use of waste clay brick as an ASR suppressing agent. The experimental work was designed to compare ground clay brick (GCB) with the other conventional pozzolans (namely a Class F fly ash and a natural pozzolan). The results showed that the GCB produced from waste clay brick has the potential to improve mortar durability: it reduces the ASR expansion, significantly helps to maintain the strength affected by the deleterious reaction, and improves the chloride permeability. Its performance is highly comparable to the natural pozzolan. These research results are significant in the countries having major brick/tile manufacturing industry (such as Turkey) and limited access to high quality SCM. Further research is needed to evaluate the potential for concrete application. An experimental program considering the use of GCB as a concrete component is in progress and the results will be published.

Acknowledgement

The writers would like to thank Professor Goncuoglu from the Department of Geological Engineering at the Middle East Technical University, Ankara for the petrographic analysis of the perlite aggregate.

References

Ahmed, T., Burley, E., Rigden, S., and Abu-Tair, AI. (2003). "The effect of alkali reactivity on the mechanical properties of concrete." *Constr. Build. Mater.*, 17(2), 123–144.

Balendran, R.V., and Martin-Buades, W.H. (2000). "The influence of high temperature curing on the compressive, tensile and flexural strength of pulverized fuel ash concrete." *Build. Environ.*, 35(5), 415–423.

Baronio, G., and Binda, L. (1997). "Study of pozzolanicity of some bricks and clays." *Constr. Build. Mater.*, 11(1), 41–46.

Barringer, W.L. (1999). "Using accelerated test methods to specify admixtures to mitigate alkali-silica reactivity." *Cement Concrete Aggr.*, 21(2), 165–172.

Bektas, F., Turanli, L., and Monteiro, P.J.M. (2004). "A preliminary study of the efficiency of crushed brick blended cement in reducing ASR." *Proc., 12th International Conference on Alkali-Aggregate Reaction*, CANMET/ACI, Beijing, China, 479–482.

Berube, M.A., and Duchesne, J., Chouinard, D. (1995). "Why the accelerated mortar bar method ASTM C1260 is reliable for evaluating the effectiveness of supplementary cementing materials in suppressing expansion due to alkali-silica reaction." *Cement Concrete Aggr.*, 17(1), 26–34.

Berube, M.A., Dorion, J.F., Duchesne, J., Fournier, B., and Vezina, D. (2003). "Laboratory and field investigations of the influence of sodium chloride on alkali-silica reactivity." *Cement Concrete Res.,* 33(1), 77–84.

Bleszynski, RF.., and Thomas, M.D.A. (1998). "Microstructural studies of alkali-silica reaction of fly ash concrete immersed in alkaline solutions." *Adv. Cem. Based Mater.*, 7(2), 66–78.

Chen, H.J., Yen, T., and Chen, K.H. (2003). "Use of building rubbles as recycled aggregates." *Cement Concrete Res.,* 33(1), 125–132.

De Brito, J., Pereira, A.S., and Correira, J.R. (2005). "Mechanical behaviour of non-structural concrete made with recycled ceramic aggregates." *Cement Concrete Comp.*, 27(4), 429–433.

Feldman, R., Prudencio, Jr. L.R., and Chan, G. (1999). "Rapid chloride permeability test on blended cement and other concretes: correlations between charge, initial current and conductivity." *Constr. Build. Mater.*, 13(3), 149–154.

Heikal, M. (2000). "Effect of temperature on the physico-mechanical and mineralogical properties of Homra pozzolanic cement pastes." *Cement Concrete Res.,* 30(11), 1827–1834.

Jones, A.E.K, and Clark, L.A. (1998). "The effects of ASR on the properties of concrete and the implications for assessment." *Eng. Struct.*, 20(9), 785–791.

Julio-Betancourt, G.A., and Hooton, R.D. (2004). “Study of the Joule effect on rapid chloride permeability values and evaluation of related electrical properties of concretes.” *Cement Concrete Res.*, 34(6), 1007–1015.

Katayama, T., Tagami, M., Sarai, Y., Izumi, S., and Hira, T. (2004). “Alkali-aggregate reaction under the influence of deicing salts in the Hokuriki district, Japan.” *Mater. Charact.*, 53(2-4), 105–122.

Khalaf, F.M., and DeVenny, A.S. (2005). “Properties of new and recycled clay brick aggregates for use in concrete.” *J. Mater. Civil Eng.*, 17(4), 456–464.

Khaloo, A.R. (1994). “Properties of concrete using crushed clinker brick as coarse aggregate.” *ACI Mater. J.*, 91(2), 401–407.

Khatib, J.M. (2005). “Properties of concrete incorporating fine recycled aggregate.” *Cement Concrete Res.,* 35(4), 763–769.

Mehta, P.K. (1984) “Chapter 6 – Mineral admixtures.” *Concrete admixtures handbook: Properties, science, and technology*, Noyes, Park Ridge, NJ.

Moropoulou, A., Cakmak, A.S., Biscontin, G., Bakolas, A., and Zendri, E. (2002). “Advanced Byzantine cement based composites resisting earthquake stresses: the crushed brick/lime mortars of Justinian's Hagia Sophia.” *Constr. Build. Mater.*, 16(8), 543–555.

O'Farrell, M., Wild, S., and Sabir, B.B. (1999). “Resistance to chemical attack of ground brick-PC mortar: Part I. Sodium sulphate solution.” *Cement Concrete Res.,* 29(11), 1781–1790.

O'Farrell, M., Wild, S., and Sabir, B.B. (2000). “Resistance to chemical attack of ground brick–PC mortar: Part II. Synthetic seawater.” *Cement Concrete Res.,* 30(5), 757–765.

O'Farrell, M., Wild, S., and Sabir, B.B. (2001). “Pore size distribution and compressive strength of waste clay brick mortar.” *Cement Concrete Comp.*, 23(1), 81–91.

Poon, C.S., and Chan, D. (2006). “Paving blocks made with recycled concrete aggregate and crushed clay brick.” *Constr. Build. Mater.*, 20(8), 569–577.

Rangaraju, P.R., Sompura, K.R., Olek, J., Diamond, S., and Lovell, J. (2005). “Potential for development of alkali-silica reaction in the presence of airfield deicing chemicals.” *Proc., 8th Int. Conf. on Concrete Pavement,* ISCP, Colorado Springs, Colo., 1269–1289.

Shi, C. (2004). “Effect of mixing proportions of concrete on its electrical conductivity and the rapid chloride permeability test (ASTM C1202 or AASHTO T277) results.” *Cement Concrete Res.*, 34(3), 537–545.

Thomas, M.D.A., Matthews, J.D., and Haynes, C.A. (1989). “The effect of curing on the strength and the permeability of PFA concrete.” *Proc., 3rd Int. Conf. on Fly Ash, Silica Fume, Slag, and Natural Pozzolans in Concrete*, CANMET/ACI, Trondheim, Norway, 191–217.

Turanli, L., Bektas, F., and Monteiro, P.J.M. (2003). “Use of ground clay brick as a pozzolanic material to reduce the alkali–silica reaction.” *Cement Concrete Res.,* 33(10), 1539–1543.

Turker, P., Erdogan, B., Katnas, F., and Yeginobali, A. (2004). *Classification and properties of fly ashes in Turkey*, Turkish Cement Manufacturers Association Ankara, Turkey (in Turkish).

Uzal, B., and Turanli, L. (2003). “Studies on blended cements containing a high volume of natural pozzolans.” *Cement Concrete Res.,* 33(11), 1777–1781.

World Business Council for Sustainable Development (WBSCD). (2005). “The cement sustainability initiative progress report.”, <http:// www.wbcsdcement.org/>.

List of Figures

List of Tables

Table 1. Chemical Composition of Cementitious Materials

Composition (%)	**OPC**	**FA**	**NP**	**GCB**
SiO_2	19.94	59.18	62.02	62.70
Al_2O_3	5.34	22.68	15.46	17.10
Fe_2O_3	3.72	6.44	3.60	6.84
(SiO_2+Al_2O_3+Fe_2O_3)	–	88.30	81.08	86.64
CaO	63.02	4.28	4.64	3.94
MgO	2.44	3.07	2.98	2.25
SO_3	2.95	0.49	0.19	0.84
Na_2O	0.58	0.09	2.06	0.58
K_2O	0.77	0.60	1.37	1.23
Na_2O_{eq}	1.09	0.48	2.96	1.39
Loss on Ignition	1.02	2.40	5.18	2.67

Note: OPC=ordinary portland cement; FA=fly ash; NP=natural pozzolan; GCB=ground clay brick.

Table 2. Flexural Strengths of 40 × 40 ×160 mm Mortar Prisms

	In water at room temperature (days)			**In 1 N NaOH at 80^{o}C (days)**		
	7	**28**	**91**	**7**	**28**	**91**
OPC	7.1	8.8	9.2	6.6	2.9	2.1
20%FA	7.4	8.8	9.5	10.7	6.3	3.2
35%FA	6.0	8.3	10.2	11.1	7.8	6.8
20%NP	7.8	8.2	9.5	10.6	5.5	3.1
35%NP	6.3	8.0	9.0	10.9	5.9	4.1
20%GCB	8.0	8.8	9.2	9.2	7.2	3.9
35%GCB	7.9	8.6	9.5	8.9	7.7	5.3
20%FA+15%NP	6.0	8.2	9.1	10.6	7.3	3.8
20%FA+15%GCB	6.3	8.6	9.2	10.3	8.1	5.0

Note: Values are in MPa.

Table 3. Compressive Strengths of 50 mm Mortar Cubes

	In water at room temperature (days)			In 1 N NaOH at 80°C (days)		
	7	**28**	**91**	**7**	**28**	**91**
OPC	38.9	42.2	45.9	36.1	32.4	35.4
20%FA	36.9	40.7	48.7	45.7	37.1	36.3
35%FA	31.0	35.6	48.5	35.1	39.3	33.0
20%NP	30.3	39.4	41.9	38.0	39.5	36.8
35%NP	25.6	31.7	42.5	35.1	39.2	35.8
20%GCB	31.7	43.2	46.1	35.8	37.2	40.3
35%GCB	27.0	34.3	41.2	33.3	34.8	36.3
20%FA+15%NP	27.3	32.8	37.8	33.9	37.5	32.5
20%FA+15%GCB	28.0	31.0	41.3	36.1	34.9	31.7

Note: Values are in MPa.

Table 4. Effect of ASR Curing on the Mortar Strengths – NaOH Curing to Water Curing Ratio

	Flexural Strength (days)			Compressive Strength (days)		
	7	**28**	**91**	**7**	**28**	**91**
OPC	0.93	0.33	0.23	0.93	0.77	0.77
20%FA	1.44	0.72	0.34	1.24	0.91	0.75
35%FA	1.85	0.93	0.67	1.13	1.10	0.68
20%NP	1.36	0.67	0.33	1.26	1.00	0.88
35%NP	1.72	0.74	0.46	1.37	1.24	0.84
20%GCB	1.15	0.81	0.43	1.13	0.86	0.87
35%GCB	1.13	0.90	0.55	1.23	1.01	0.88
20%FA+15%NP	1.75	0.90	0.41	1.24	1.14	0.86
20%FA+15%GCB	1.64	0.94	0.54	1.29	1.13	0.77

Table 5. Rapid Chloride Permeability (ASM C1202)

	Charge (C)	
	28-day at 23^{o}C	**7-day at 80^{o}C**
OPC	3,859	10,117
35%FA	1,264	544
35%NP	2,074	9,64
35%GCB	1,631	1,356
20%FA+15%NP	1,807	702
20%FA+15%GCB	1,628	721

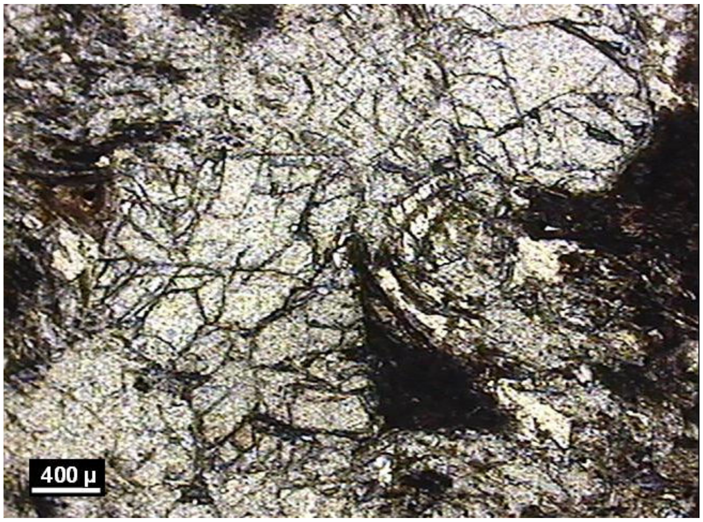


Fig.1. Microstructure of the perlite aggregate – pores and spherical cracks provide a large surface area for the aggregate to contact alkali ions (Thin section – optical microscope)

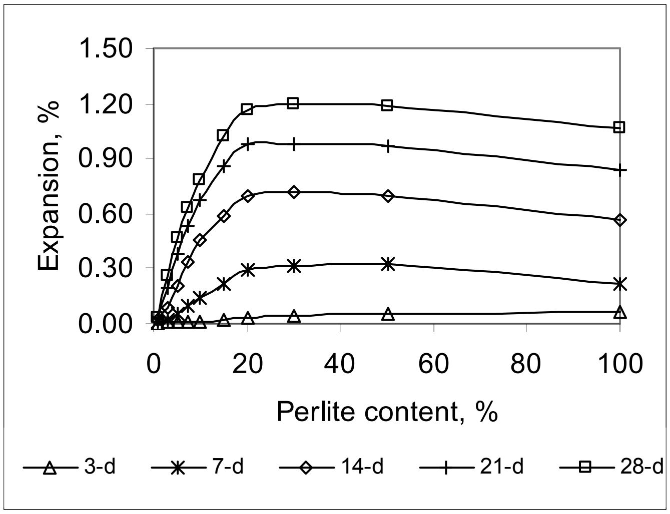


Fig.2. Pessimum behavior of perlite aggregate

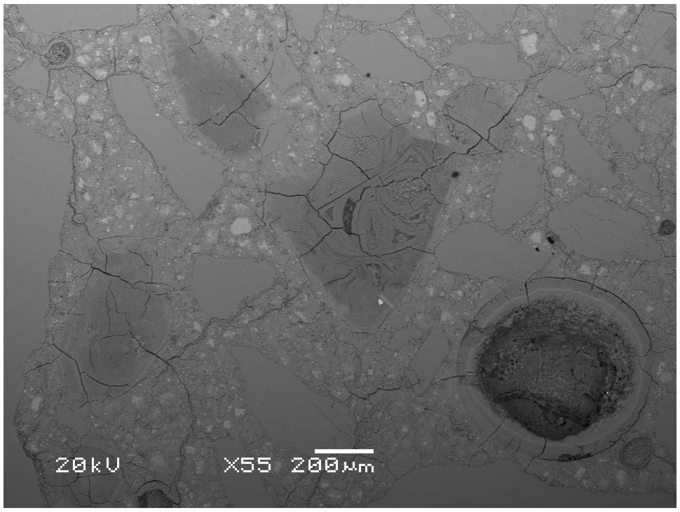


Fig.3. Internal deterioration of control sample after 1 year of NaOH exposure – BSE image (cracks are seen in paste and perlite particles; however, most limestone particles remain intact)

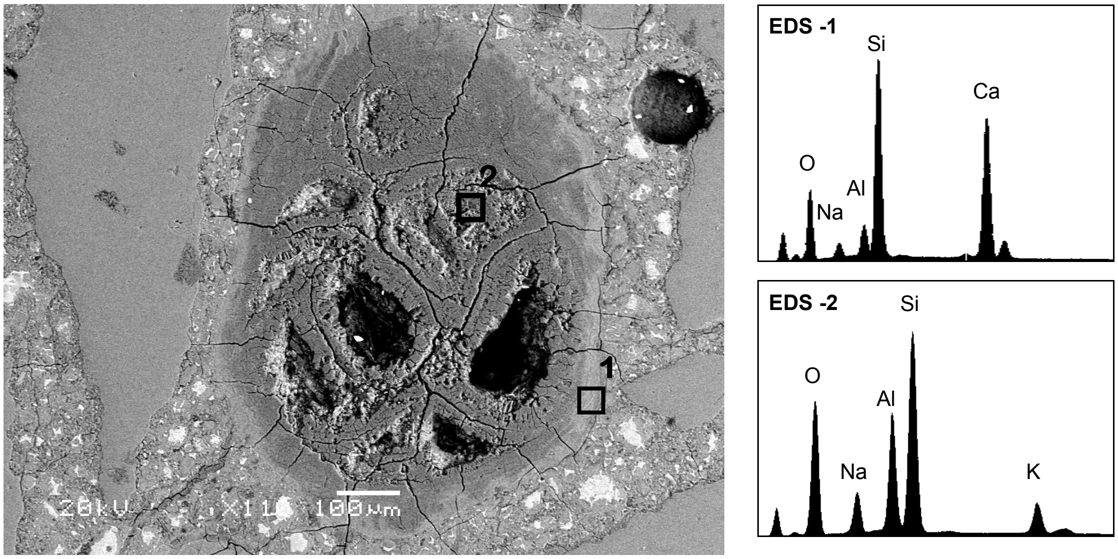


Fig.4. Perlite particle transformed into ASR gel – BSE image (radial cracks and calcium-rich ASR rim are seen)

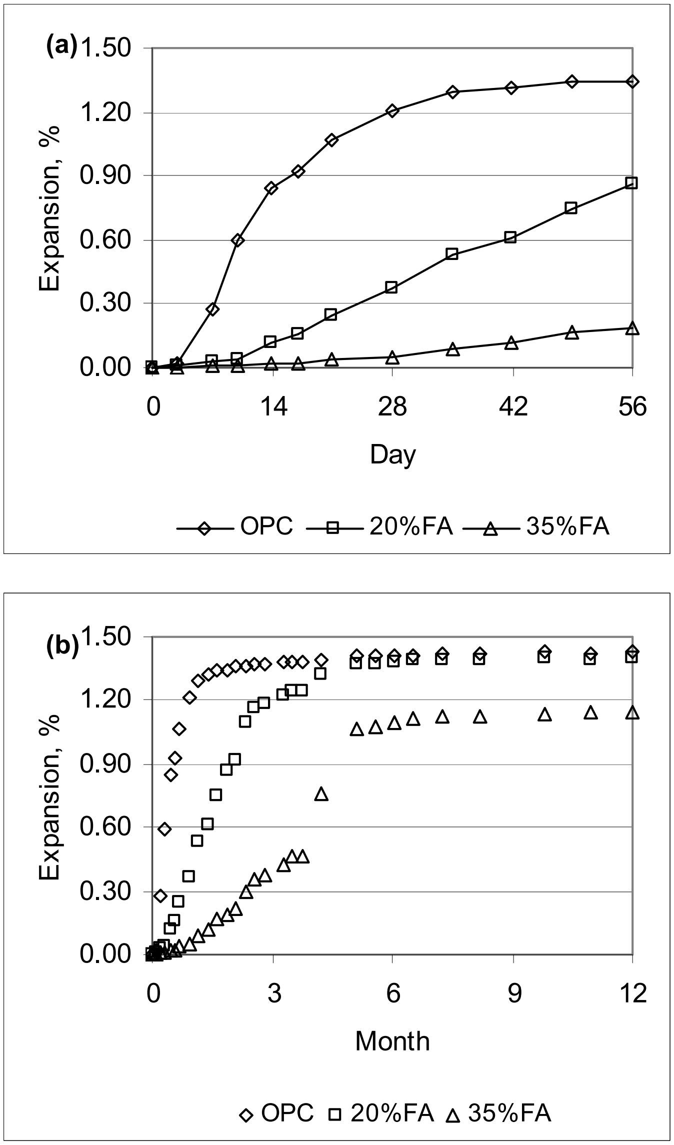


Fig. 5. ASR expansion curves of fly ash samples up to: (a) 56 days; (b) 1 year

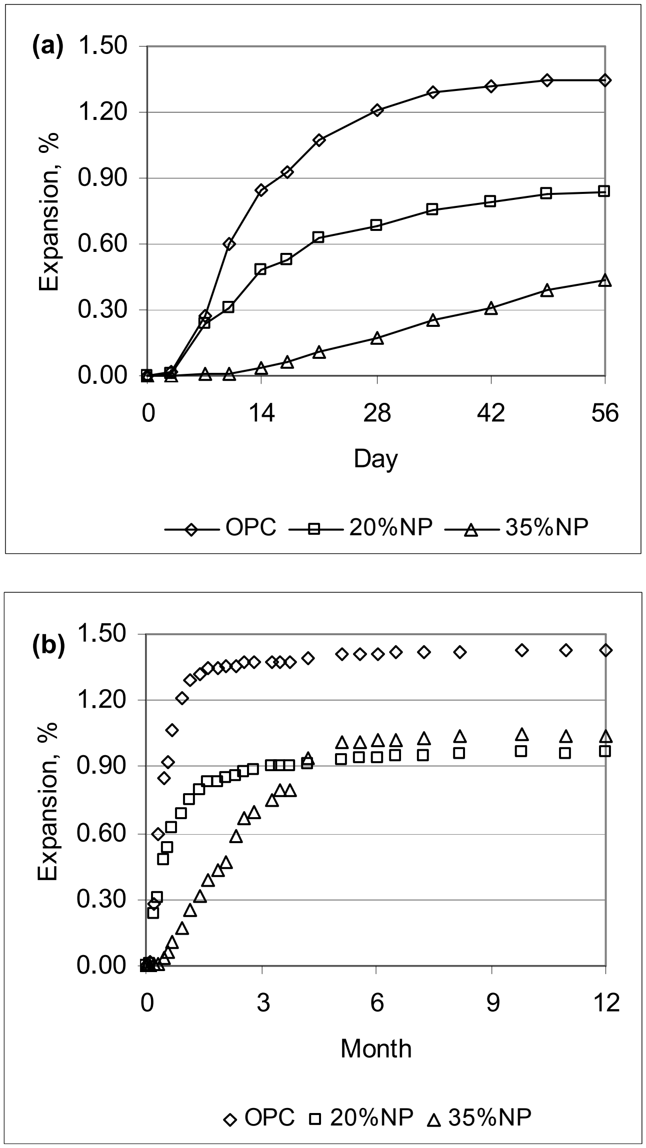


Fig. 6. ASR expansion curves of natural pozzolan samples up to: (a) 56 days; (b) 1 year

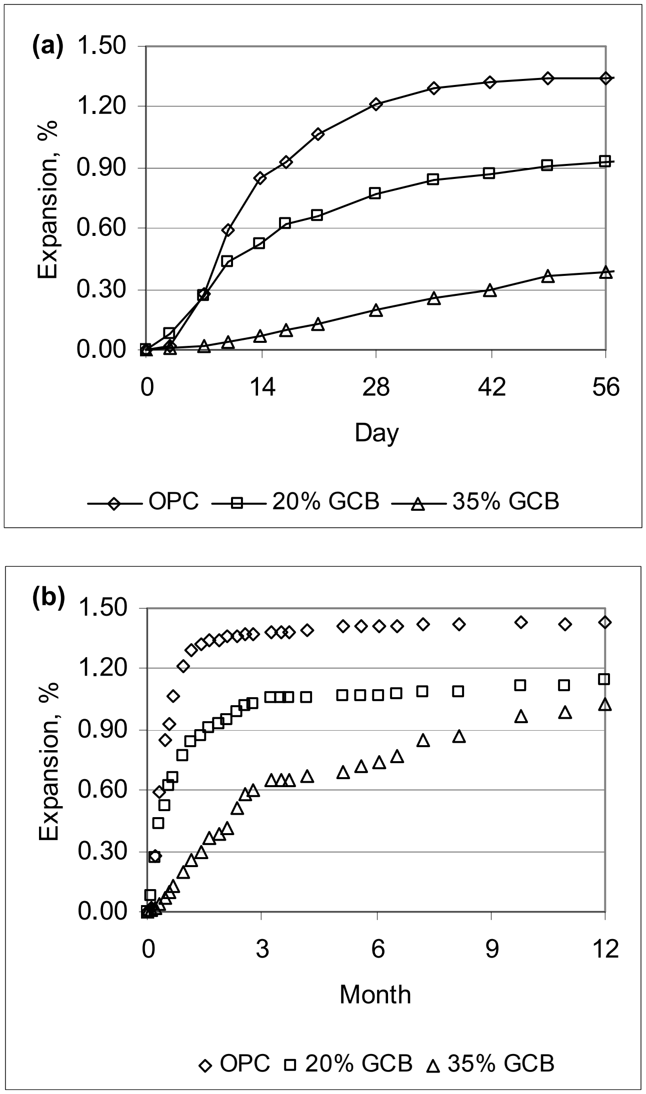


Fig. 7. ASR expansion curves of ground clay brick samples up to: (a) 56 days; (b) 1 year

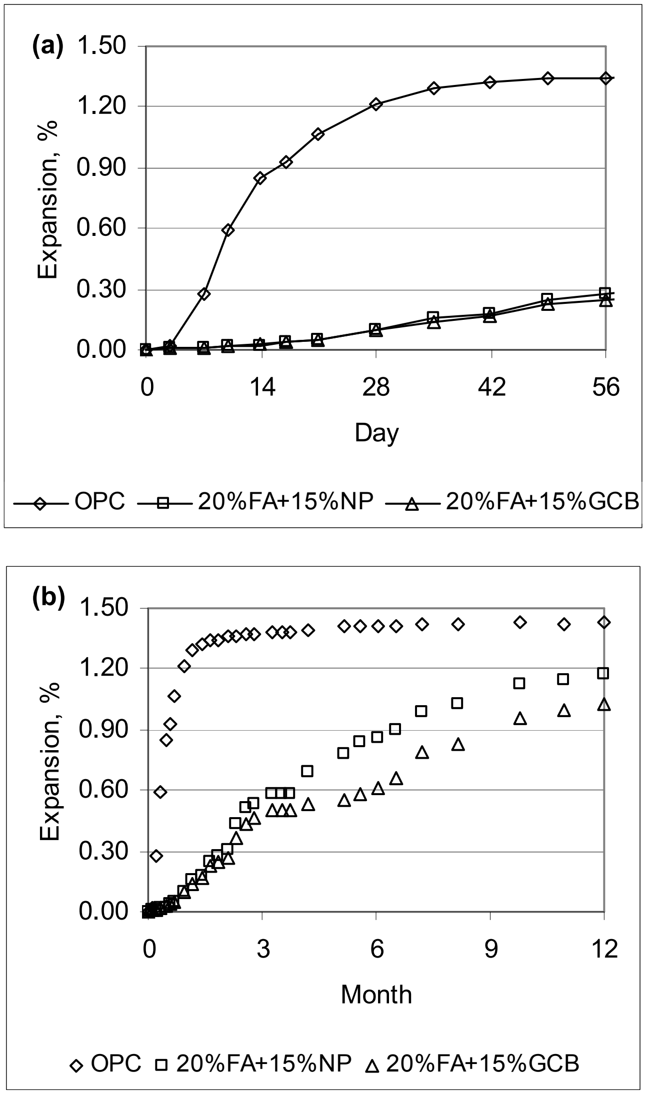


Fig. 8. ASR expansion curves of ternary binder samples up to: (a) 56 days; (b) 1 year

Chapter 5

Control of ASR Damage on Mechanical Properties of Concrete Using Ground Clay Brick

A paper to be submitted to Cement and Composites (Elsevier)

F. Bektas[1], K. Wang[2], H. Ceylan[3]

Abstract

Effectiveness of ground clay brick (GCB) in preventing or mitigating the damage on the mechanical properties of concrete due to the deleterious ASR was investigated in comparison to a Class C fly ash. Waste clay brick originated from demolished masonry was ground to cement fineness and introduced to concrete as cement replacement at levels of 15 and 25%. Mixtures with the same levels of Class C fly ash together with a control (only portland cement) were also prepared. It was found that the ground clay brick effectively reduces the ASR expansion and the associated losses in mechanical properties of concrete (e.g., flexural strength, modulus of elasticity). The performance of concrete containing GCB is superior to that of concrete containing Class C fly ash. Microscope study also revealed that the GCB reduces the quantity of ASR gel produced. As a result the waste clay brick can be effectively used in concrete to alleviate ASR related deterioration.

Keywords: Ground clay brick (GCB); ASR; Mechanical property

---

[1] Graduate student; primary researcher and author; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[2] Associate professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[3] Assistant professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

1. Introduction

Interaction of alkalis with reactive siliceous aggregate leads to deleterious alkali-silica reaction (ASR) in concrete. The reaction manifests itself as a three-dimensional array of cracking. Depending on the degree of the reaction, ASR diminishes the engineering properties of concrete (i.e. strength, stiffness). Tensile strength and modulus of elasticity of concrete are very sensitive to ASR cracking; their reductions may be up to 80%. Compressive strength of concrete is also reduced due to the ASR damage although to a lesser extent [1–5].

After grinding, clay brick obtained from brick manufacturing rejects or demolished masonry waste can be used as a pozzolanic material in concrete to improve concrete durability, such as resistances to sulfate attack and alkali-silica reaction. O'Farrell et al. [6] showed that the ground clay brick (GCB) reduced the mortar expansion and strength loss due to sulfate attack. Bektas et al. [7] demonstrated that ASR expansion could be effectively suppressed by the use of GCB, and the degradation in mechanical properties of mortar mixtures was also prevented. However, the previous research on the use of GCB mostly focused on the study of mortar, while literature concerning the utilization of GCB in concrete is very limited.

In the broader aspect, this study is the continuation of an effort to recycle waste clay brick and use the GCB as a pozzolanic material in concrete. The experimental program aimed to study the effect of finely GCB on ASR susceptible concrete mixture. Different from most previous studies, which usually evaluate only the effectiveness of selected pozzolanic materials in terms of their capacity to reduce ASR induced expansion, the present study, includes the determination of mechanical properties of concrete mixtures containing 15% and 25% of either GCB or Class C fly ash in addition to measuring the ASR expansion. Microscope study of the concrete was also carried out in an attempt to better understand the mechanisms of concrete deterioration involved.

2. Materials

The cementitious materials used in the study were a high-alkali portland cement, a finely GCB and a Class C type fly ash. The chemical compositions are provided in Table 1. The residues on 45-micron (ASTM No.325) sieve are 10.2% (with a specific gravity of 2.68) and 11.9% (2.69) for the GCB and the fly ash, respectively. GCB and Class C fly are alternately referred as pozzolanic material or supplementary cementitious material (SCM) in this paper.

Highly reactive sand from New Mexico (USA) was used as fine aggregate in the concrete mixtures. The sand contains considerable amounts of volcanic glass (4%) and chert (2%). In addition, andesite, granite and recrystallized quartz particles are also present as potentially reactive constituents. Locally available crushed oolitic limestone was utilized as coarse aggregate.

3. Experimental Program

Before concrete testing accelerated ASR testing of mortar bars was carried out as a pre-evaluation of the available materials. A total of 5 mortar mixtures—a control (no SCM) were prepared – control, B15, B25, C15 and C25, were prepared with the above-mentioned reactive fine aggregate. The ground brick mixtures were denoted as B (for brick), and similarly, the fly ash mixtures as C (for Class C fly ash). The number at the end represents the amount of replacement by cement weight. The bars, then, were subjected to accelerated ASR testing (ASTM C 1260—*Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar Bar Method)*). The fine aggregate gradation was used as received (Table 2) from the quarry instead of the standard grading given in ASTM C1260. The bars were stored in 1N sodium hydroxide (NaOH) solution at 80^{o}C and the expansions were recorded up to 28 days.

Similar to those used for the accelerated ASR mortar testing, a total of 5 concrete mixtures, control, B15, B25, C15, and C25, were prepared. The reactive fine aggregate and the non-reactive coarse limestone were used in the concrete. The mixture proportion for the control concrete is given in Table 3. The alkali content of the portland cement was adjusted to 1.5% in all concrete mixtures by adding NaOH to the mixing water.

Concrete prisms, 75×75×285 and 75×75×300 mm^3, were prepared for ASR expansion and flexural strength measurements, respectively, and cylinders, 75×150 mm^3, were cast for compressive strength and modulus of elasticity determination. The specimens were stored in insulated tanks maintained at 50^{o}C and >95% relative humidity in order to accelerate ASR. The expansion measurements were taken continuously up to 6 moths. The compressive strength (ASTM C 39—*Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*), the flexural strength (ASTM C 78—*Standard Test Method for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading)*) and the modulus of elasticity (ASTM C 469—*Standard Test Method for Static Modulus of Elasticity and Poisson's Ratio of Concrete in Compression*) tests were executed at 14, 28, 56, 91 and 180 days.

After 6 months of curing, sections were cut and polished from the concrete cylinder specimens, and examined under variable pressure scanning electron microscopy (SEM). In order to study the ASR gel formation and composition, a pyrex glass rod, having 4 mm diameter and 250 mm length, was embedded at the center of pastes (25×25×285 mm^3) made with plain portland cement and the cement with 25% GCB replacement. The pastes were cured over water at 50^{o}C for 6 months. Sections of 25×25 mm^2 in cross-section were cut from pyrex rod-paste sample and examined under SEM with energy dispersive spectrometer (EDS). In the sample preparation, propylene glycol was used for the sample cutting and polishing. First, the sections were impregnated with epoxy under vacuum and the surface was polished using successive grinding silicon-carbide papers.

4. Results and Discussion

4.1. ASR expansion of mortar samples

Expansion curves of the mortar bars stored in NaOH solution are given in Fig. 1. The plot denoted as Control-1260 resulted from an unpublished study, where the mortar made with the reactive aggregate having the required ASTM gradation (The 14-day expansion of this sample is 0.87%.). The fine aggregate used in the study had a high fraction of fine particles when compared with the standard aggregate gradation (Table 2), and it produced higher expansion. The GCB and the Class C fly ash reduced the expansion at all replacement levels. The 14-day expansions, in descending order, were 1.01, 0.80, 0.61, 0.31 and 0.12% for the control, C25, C15, B15 and B25, respectively. The results clearly demonstrate the superior performance of the GCB over the fly ash. ASTM C1260, or recently published ASTM C1567—*Standard Test Method for Determining the Potential Alkali-Silica Reactivity of Combinations of Cementitious Materials and Aggregate (Accelerated Mortar-Bar Method)*, limits the expansion as 0.10% at 14 days. Due to the gradation of the aggregate used, which provided mortar with a higher expansion than the aggregate with the standard gradation, it can be assumed that 15% GCB is not sufficient to suppress the harmful expansion and 25% GCB lies just on the limit. Similarly, the Class C fly ash is not effective even at 25%. It is known that Class C fly ashes, due to their high CaO content, are not effective in mitigating ASR. For a given reactivity, as CaO content increases, higher amounts of the fly ashes are needed to suppress the reaction. Therefore, a CaO limit is sought for use in ASR suppression: the limits are 10 and 8 %, respectively, based on the AASHTO and Canadian standards. Class C fly ash is not permitted in ASR suppression in Canada [8].

Fig. 2 gives the expansion measurements of the concrete prisms stored at 50°C over water in the sealed tank. The expansions were leveled off between 3 and 6 months. The values in the descending order were 0.235, 0.152, 0.043, 0.035 and 0.023% for the control, C15, B15, C25 and B25 concrete samples, respectively. ASTM C1293—*Standard Test Method for Determination of Length Change of Concrete Due to Alkali-Silica Reaction* recommends 0.04% expansion as the limit between deleterious and tolerable reaction. Based on this limit, B25 and C25 samples pass the criterion while B15 is just on the limit and C15 fails. Cracks could be observed visually on the control and C15 prisms. While there were no cracking, the rest of the prisms displayed spots of carbonated ASR gels on their surface which suggested ASR also took place in these samples although the expansion is minimal. The testing of the mechanical properties and microscope investigation of these samples as discussed later further revealed out the degree of degradation.

The results of concrete expansions depicted a different feature of ASR deterioration when compared to the accelerated mortar bar testing. In the concrete testing, 15% GCB fell on the limit line with no visual cracking. Unexpectedly, a relatively low level of Class C fly ash replacement (25%) was sufficient for suppressing concrete expansion. Shehata and Thomas [9] studied concrete with a wide range of fly ashes representing a considerably large array of chemical composition, and they concluded that at a given replacement level, the expansion of concrete prisms generally increased as the calcium or alkali content of the fly ash increased or as its silica content decreased. Based on Shehata and Thomas, the minimum replacement level that is required to keep the expansion below 0.04% for the fly ashes having CaO content >20% with low alkali content (<4%) is in a range of 25 to 60%. Their findings indicate that the performance of fly ashes in suppressing ASR cannot be explained solely based on the chemical composition of the cementitious material.Aggregate reactivity, mixture proportions, and curing conditions also contribute to ASR, and may affect the effectiveness of the SCM in ASR suppression.

4.2. Mechanical properties

Figs. 3–5 give the results of compressive strength, flexural strength and modulus of elasticity testing, respectively. The samples with the same amount of SCM replacement levels, together with the control sample, are presented in separate plots. Fig. 3(a), 4(a) and 5(a) show a comparison of the control and 15% SCM replacement samples while Fig.

3(b), 4(b) and 5(b) demonstrates the properties of the samples with 25% SCM replacements.

As given in Fig. 3 the compressive strength values are clustered around 35 MPa for all the mixtures regardless of the age. Strength gains from the initial measurement to the last one are 6, 20, 16, 1 and 8% for the control, B15, B25, C15 and C25, respectively. Despite the visual evidence of cracking and gel exudation on the specimen surface, the control mixture did not experience a strength loss but a slight gain. Cracking was also visible on the C15 specimens whereas the other mixtures remained intact. The compressive strength of GCB and Class C ash samples are either higher than or comparable to that of the control sample. Fig. 4 shows the flexural strength calculated as the modulus of rupture. Negative effect of ASR is clearly seen on the control and the C15 samples. These two concrete mixes had reduced flexural strength values compared to the other concrete mixes, however, C15 was still better than the control. The GCB showed superior performance in eliminating the strength loss due to ASR cracking, at both 15 and 25% replacement levels. At the same replacement level, the GCB samples achieved higher flexural strength values compared to the Class C fly ash samples. In addition, the results are in well agreement with the expansion measurements. It was evident that the expansion and associated cracking was reflected on the flexural strength. Furthermore, the concrete experienced considerable expansion (control and C15), showed a distinctive behavior that after a drop in flexural strength a recovery occurs at the later ages. The control concrete reached its lowest value at 56 days and then, showed a gradual increase in flexural strength. Similarly the flexural strength of C15 increased after a drop from 14 to 28 days. Ahmed et al. [4] also reported similar behavior. The phenomenon was explained on the basis of on-going hydration dominates the detrimental effect of ASR which slows down or stops. The presence of SCM, Class C fly ash in this case, might help a faster recovery. The results of modulus of elasticity testing are plotted in Fig 5. The overall appearance is identical to the flexural strength plot. Control and C15 carries the negative effect of ASR whereas B15, B25 and C25 are unaffected. The GCB samples outperformed the fly ash ones at a given replacement level. 15% GCB sample showed the best performance in the tested samples. The similar recovery behavior was also observed in the modulus of elasticity of the detrimentally expanded concretes—control and C15.

In summary, the testing of mechanical properties of ASR prone concrete mixtures has proved the beneficial effect of SCMs, GCB and fly ash, in mitigating ASR. The ground brick used in the study showed superior performance when replaced cement at the 15 and 25% cement-replacement levels. In addition, it outperformed Class C fly ash. The tests showed that the expansion and associated cracking due to ASR is significantly reflected on the flexural strength and the modulus of elasticity as reduction. On the other hand, the compressive strength was less sensitive to the ASR-associated cracking.

4.3. Microscope investigation

Microscopic investigation was performed for a better understanding of the role of GCB in ASR. In general, observations at the micro level confirm the visual condition of the specimens: the control concrete was heavily cracked and showed evidence of reaction products whereas the effect of ASR on the SCM incorporated mixes was minimal. Figs. 6 and 7 show the backscattered electron (BSE) images from the control sample. The reaction product, ASR gel, was detected filling the cracks, running along the aggregate boundaries, or lining the edge of air voids. There was evidence of the gel that was rich in calcium and low in alkali. Wang and Gillot [10] suggested that there is a cycle of cation exchange of alkali for calcium as concrete ages. In addition, Chatterji [11] claimed that alkali-silica gel is more fluid and mobile when calcium content is low. With a consideration on the age of the specimen, the observations in this study suggest that the alkali-silica gel exchanged its alkali ions with calcium from the surrounding cement paste. The resulting complex is a calcium-silicate gel which may be similar to cement hydration products and more stable than initial alkali-rich gel. The partial recovery observed in the flexural and modulus of elasticity testing can be explained on basis of this observation: after expansion and cracking occurs initially fluid alkali-rich gel flows through the cracks and fills them, then, through alkali-calcium exchange it becomes more viscous, more stable and bonded to surrounding paste acting as a grout.

Some carbonated gel was spotted on the concrete prisms containing GCB. Microscope observations proved the existence of ASR in the GCB concrete. The number of the sites detected with ASR gel was lower compared to the control sample. No sign of cracking was observed in the control mixture. Fig. 8 shows a reaction site with the gel. The gels were accumulated in the voids: as Bleszynski and Thomas [12] postulated the gel might be fluid and capable of dispersing through cement matrix not causing expansion. Class C fly ash concrete provided similar observations as in GCB mixture except that there was abundance of ettringite formation in this mix. Fig. 9 shows ettringite crystals that are located in an air void. This finding is in well agreement with the literature, which states the co-existence of ettringite with high-calcium content fly ash [13,14].

The fine aggregate used in the study was composed of various reactive constituents. For the investigation on ASR gel chemistry, therefore, the study was carried out using pyrex glass in order to have a homogeneous reactive medium. The reaction took place both in the control and the GCB samples, however, the quantity of gel in the control was significantly higher. Fig. 10 shows the x-ray mappings of the reaction rim around the pyrex taken from the control and the GCB (25% replacement) samples. Associated BSE images are also presented. The gels are clearly identifiable in the figures and both gels show a distinctive featuring of potassium: the potassium is highly concentrated in the ASR gel while the sodium is relatively scattered in the surrounding paste as well as in the gel. The results suggest that the potassium in the paste is selectively attracted to the reaction site namely the pyrex surface.

EDS on the pyrex showed it is mostly composed of silica (~85%) with the presence of sodium (~4–5%) and aluminum (~2–3%). EDS point analyses were done along the gels. Number of the data points taken is proportional to the quantity of the gel: 12 and 8 points for the control and the GCB samples, respectively. The results presented as oxide are given in Table 4. The difference form 100% is attributed to the water. The results demonstrate a difference between the alkali (as sodium equivalent) and calcium contents between the gel compositions: the calcium is higher and the potassium is lower in the plain portland cement paste compared to the GCB one. Unpaired t-test was run for the sodium equivalent and the calcium contents and the difference was found to be statistically significant: two-tailed P value equals to 0.021 and 0.029 for the control and the ground brick, respectively. Bleszynski and Thomas [12] studied the ASR gel composition in the concrete sections containing plain portland cement and 40% fly ash and postulated that the high-calcium viscous gel found in the plain portland cement samples produces expansive forces whereas the alkali-rich gels in fly ash samples are less viscous and do not cause expansion. They attributed the role of calcium acting as a semi-permeable membrane imprisoning alkali ions in reaction sites or alternately calcium gel itself is more expansive. Bektas et al. [15] proved that GCB reduces the portlandite content of paste. Consumption of $Ca(OH)_2$, which reduces calcium content in concrete, could help in the low-calcium gel formation, subsequently resulting in less expansion. On the other hand, it is worth to note that the range of the measured sodium equivalents and the calcium contents were in a relatively wide band: the coefficients of variations for calcium and sodium equivalent are 34 and 11%, and 17 and 17% for the control and the brick samples, respectively. Therefore, it might be misleading to draw a solid conclusion based on the gel chemistry but this area merits further attention. The ASR associated phenomena present a complex structure and cannot be explained based on a single mechanism. Nonetheless, it is evident that at a given age the quantity of gel produced is less in the presence of GCB.

5. Conclusions

Based on the results of this experimental study following conclusions can be drawn:

- Both mortar and concrete expansion tests showed that GCB reduces the expansion in the presence of highly reactive sand when used as a cement replacement at the 15 and 25% levels, GCB is highly effective in the expansion reduction when compared with the Class C fly ash used.
- GCB mitigates the negative effects of ASR induced cracking on the mechanical properties of concrete, particularly, flexural strength and modulus of elasticity.
- Microscope study confirmed the expansion measurements and mechanical testing that GCB concrete had minimal deterioration. The amount of gel produced in concrete containing GCB is less compared to that in the control (no GCB) concrete. The study with the pyrex rod-cementitious pastes confirmed this finding. The GCB leads to the production of an alkali-rich and calcium low alkali-silica gel which may be less expansive.
- This study proved that ground clay brick obtained from waste material can be used as a pozzolanic material in concrete so as to mitigate ASR associated degradation (i.e., expansion, strength and stiffness loss) effectively.

References

[1] Swamy RN, Al-Asali MM. Engineering properties of concrete affected by alkali-silica reaction. ACI Mater J 1985;85(5): 367–374.

[2] Rigden SR, Majlesi Y, Burley E. Investigation of factors influencing the expansive behavior, compressive strength and modulus of rupture of alkali-silica reactive concrete using laboratory concrete mixes. Mag Concrete Res 1995;47(170): 11–21.

[3] Jones AEK, Clark LA. The effects of ASR on the properties of concrete and the implications for assessment. Eng Struct 1998;20(9): 785–791.

[4] Ahmed T, Burley E, Rigden S, Abu-Tair AI. The effect alkali reactivity on the mechanical properties of concrete. Constr Build Mater 2003;17: 123–144.

[5] Marzouk H, Langdon S. The effect of alkali-aggregate reactivity on the mechanical properties of high and normal strength concrete. Cement Concrete Comp 2003;25(4-5): 549–556.

[6] O'Farrell M, Wild S, Sabir BB. Resistance to chemical attack of ground brick-PC mortar: Part I. Sodium sulphate solution. Cement Concrete Res 1999;29(11): 1781–90.

[7] Bektas F, Turanli L, Wang K, Ceylan H. Comparative performance of ground clay brick in mitigation of alkali-silica reaction. J Mater Civil Eng, in press.

[8] Malvar LJ, Cline GD, Burke DF, Rollings R, Sherman TW, Greene JL. Alkali-silica reaction mitigation: state of the art and recommendations. ACI Mater J 2002;99(5): 480–489.

[9] Shehata MH, Thomas MDA. The effect of fly ash composition on the expansion of concrete due to alkali-silica reaction. Cement Concrete Res 2000;30(7): 1063–1072.

[10] Wang H, Gillott JE. Mechanisms of alkali-silica reaction and significance of calcium hydroxide. Cement Concrete Res 1991;21(4): 647–654.

[11] Chatterji S. The role of $Ca(OH)_2$ in the breakdown of Portland cement concrete due to alkali-silica reaction. Cement Concrete Res 1979;9(2): 185–188.

[12] Bleszynski RF, Thomas MDA. Micstructural studies of alaklai-silica reaction in fly ash concrete immersed in alkaline solution. Adv Cem Based Mater 1998;7(2): 66–78.

[13] Zhao FQ, Ni W, Wang HJ, Liu HJ. Activated fly ash/slag blended cement. Resour Conserv Recy 2007;52(2): 303–313.

[14] Bektas F. Preventive measures against alkali-silica reaction. MSc Thesis. Ankara, Middle East Technical University, 2002.

[15] Bektas F, Wang K, Ceylan H. Use of ground brick as a pozzolanic material in concrete. Build Environ, submitted.

List of Figures

List of Tables

Table 1. Chemical composition of the cementitious materials

Composition (%)	**Portland Cement**	**Ground Clay Brick**	**Class C fly Ash**
CaO	61.1	0.8	25.9
SiO_2	19.5	69.9	35.4
Al_2O_3	5.0	15.4	18.9
Fe_2O_3	2.4	6.8	7.1
MgO	2.3	1.6	-
SO_3	4.4	0.1	2.4
Na_2O_{eq}	1.1	2.8	1.1
Loss on ignition	2.3	0.2	0.4

Table 2. Fine aggregate gradation for the accelerated mortar bar testing

Retained on	**Mass (%)**	**ASTM C1260**
4.75 mm (ASTM No.4)	4	-
2.36 mm (ASTM No.8)	12	10
1.18 mm (ASTM No.16)	4	25
600 µm (ASTM No.30)	17	25
300 µm (ASTM No.50)	36	25
150 µm (ASTM No.100)	21	15
75 µm (ASTM No.200)	5	-
pan	1	-

Table 3. Proportions for the control mixture

Portland cement (kg/m^3)	390
Water (kg/m^3)	195
w/cm	0.50
Reactive sand (kg/m^3)	775
Limestone aggregate (kg/m^3)	925

Table 4. Chemical composition of ASR gel

	Control			**25% GCB**		
% weight oxide	Average	Standard Deviation	Range	Average	Standard Deviation	Range
SiO_2	49.70	1.82	47.26–54.60	50.39	1.04	48.76–57.93
Al_2O_3	4.25	2.98	0.81–8.22	5.10	2.36	2.06–7.33
Fe_2O_3	0.12	0.10	0.00–0.30	0.22	0.09	0.11–0.33
MgO	0.27	0.08	0.13–0.40	0.40	0.13	0.27–0.65
CaO	12.21	4.09	6.41–17.43	8.10	1.36	6.34–10.15
Na_2O	5.16	1.06	2.78–6.97	7.37	3.55	3.77-12.78
K_2O	15.39	2.01	12.81–18.63	17.01	1.17	15.24–18.33
SO_3	0.33	0.14	0.15–0.57	0.27	0.07	0.17–0.40
Na_{eq}	15.25	1.73	13.19–17.52	18.53	3.08	15.17–23.42
Sum	87.41	2.27	84.29–92.78	88.87	1.01	87.03–89.94

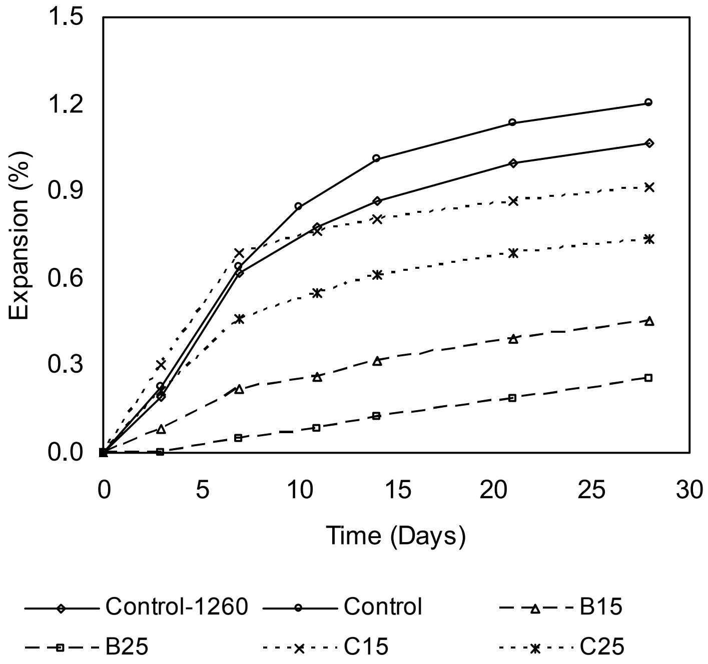


Fig.1. Mortar-bar expansions in 1N NaOH solution at 80°C.

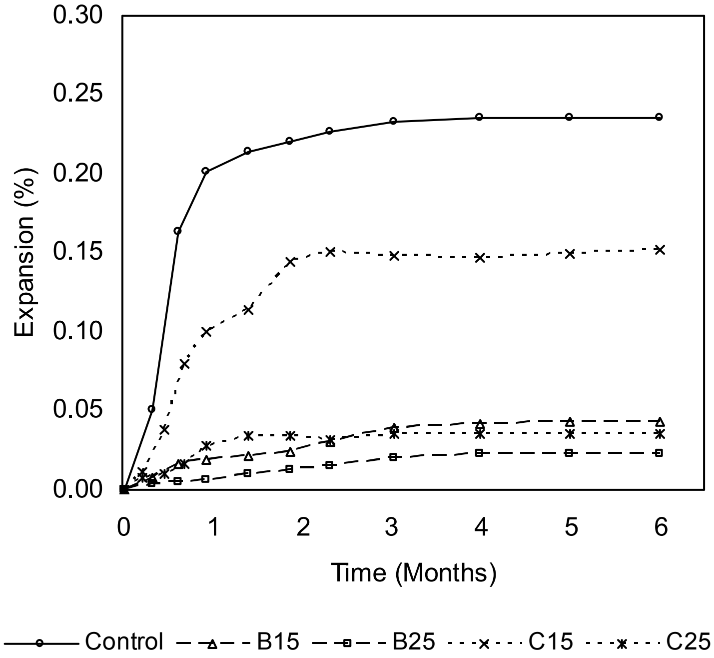


Fig. 2. Concrete prism expansion at 50^{o}C over water.

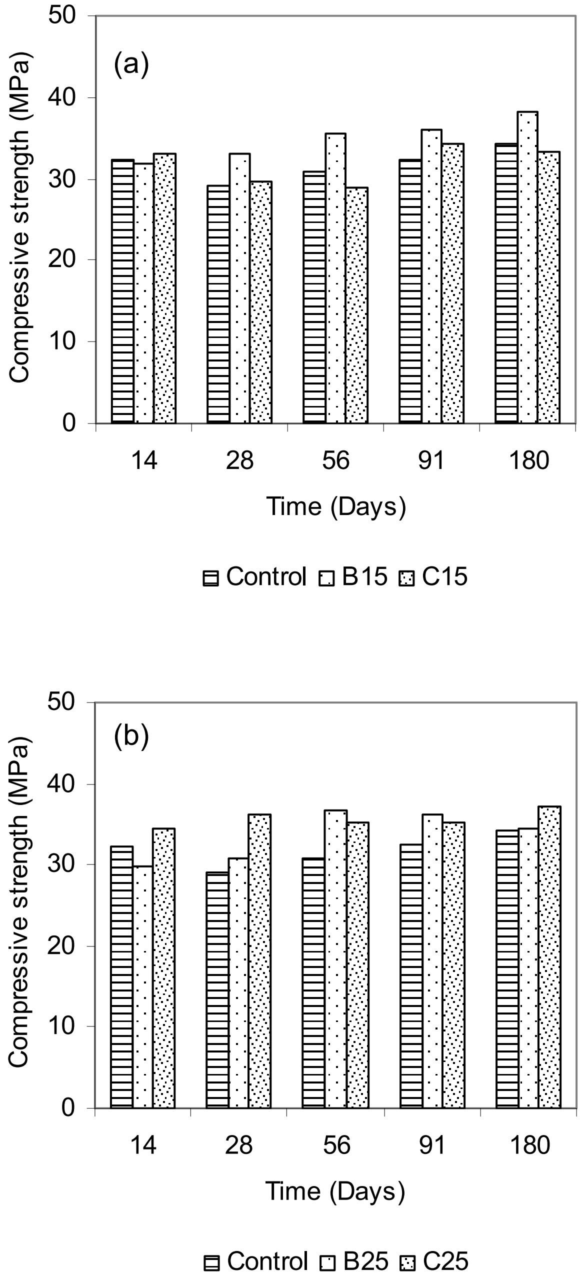


Fig. 3. Comparison of compressive strength with time.

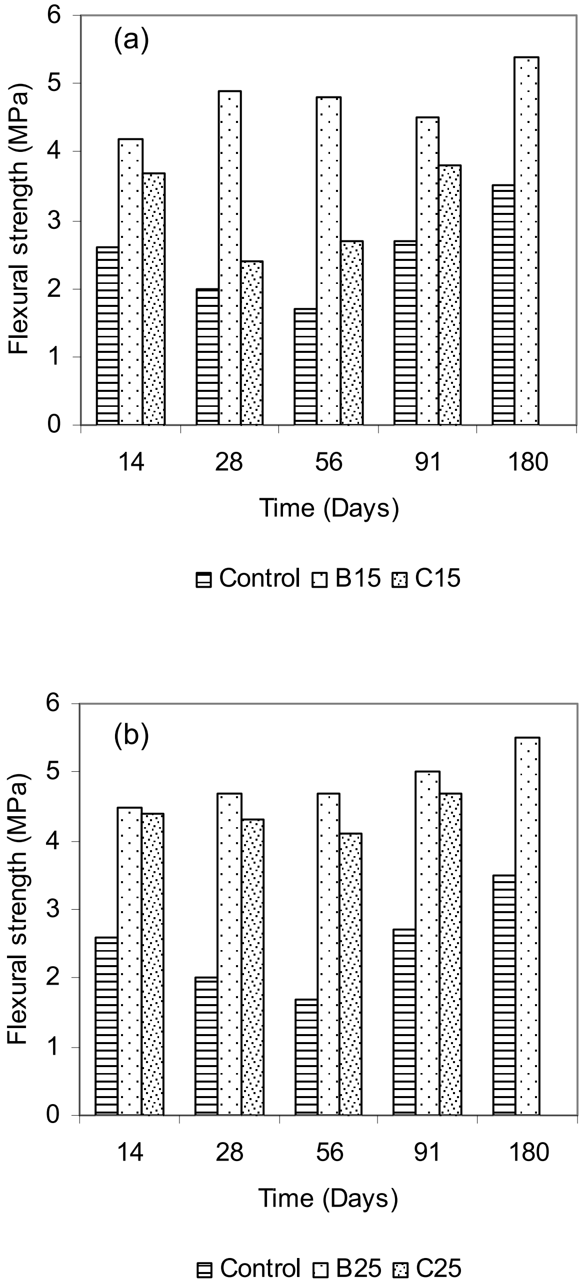


Fig. 4. Comparison of flexural strength with time.

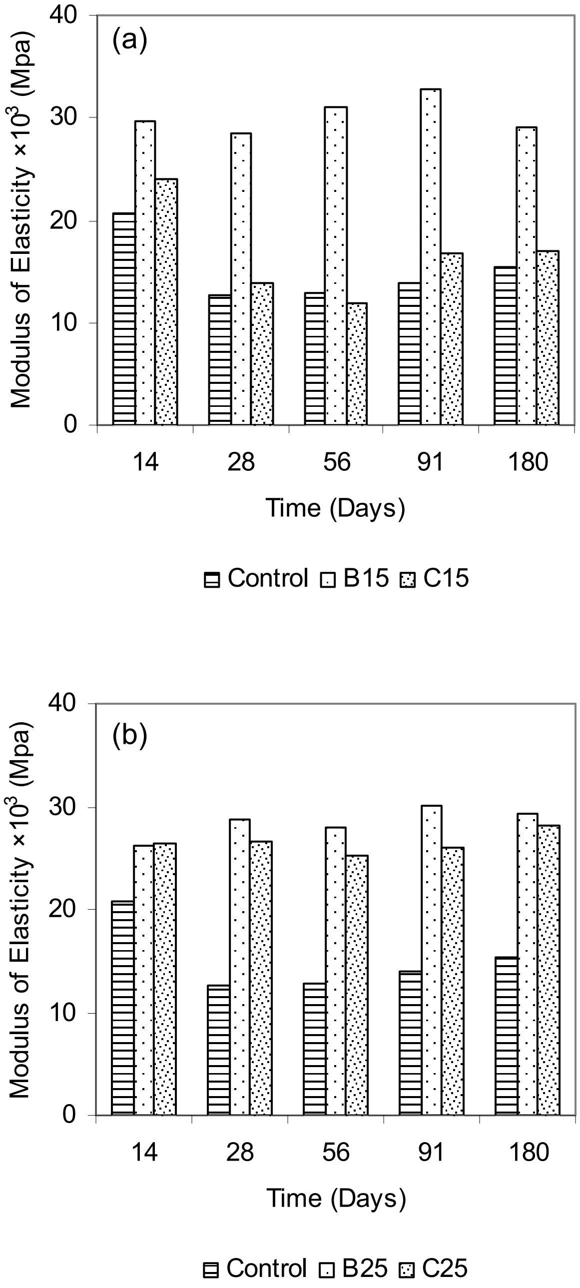


Fig. 5. Comparison of modulus of elasticity with time.

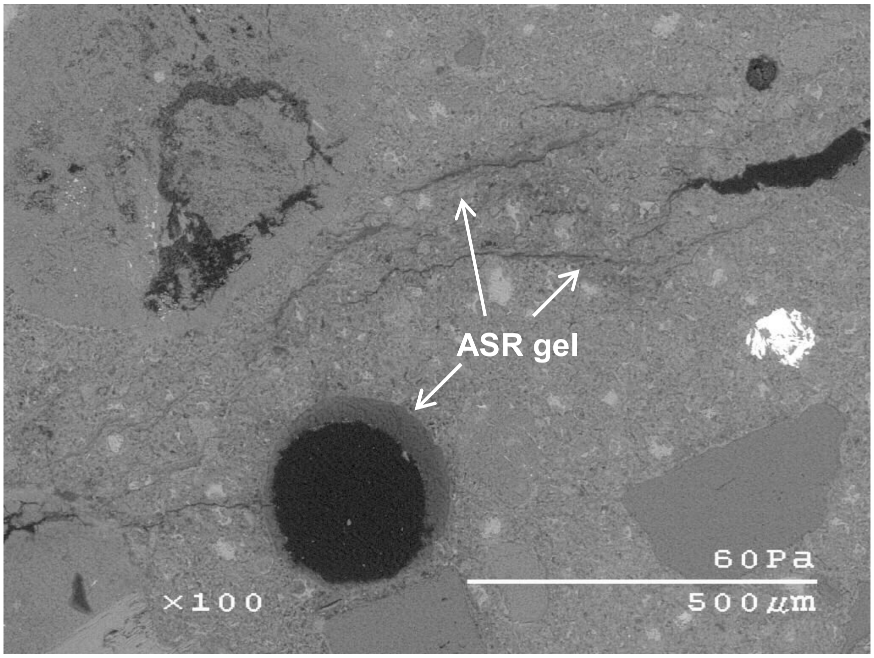


Fig. 6. ASR gel lining the air void and filling the cracks (*Point analysis on the gel in the void:* SiO_2= 42.68; Al_2O_3= 2.74; Fe_2O_3= 0.57; MgO= 0.18; CaO= 41.12; Na_2O= 0.94; K_2O= 0.83; SO_3= 0.75).

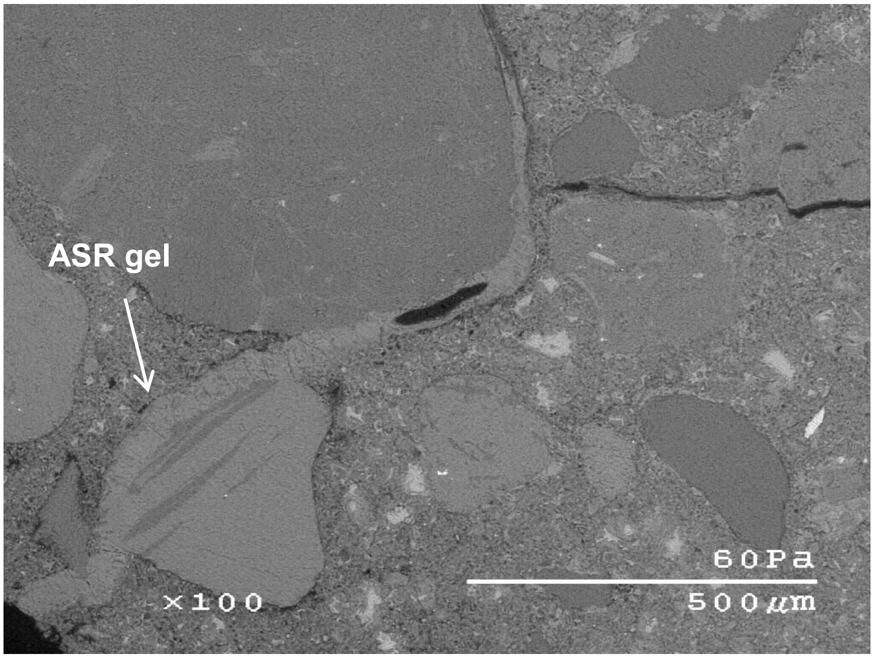


Fig. 7. ASR gel running along the aggregate boundaries (*Point analysis:* SiO_2= 41.05; Al_2O_3= 3.65; Fe_2O_3= 0.43; MgO= 0.40; CaO= 33.31; Na_2O= 2.86; K_2O= 2.34; SO_3= 0.42).

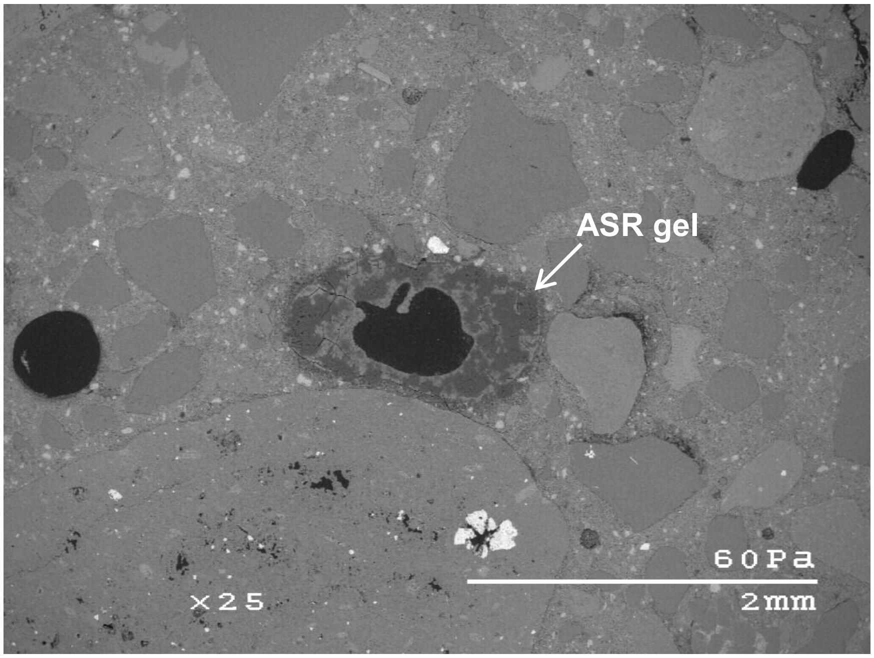


Fig. 8. ASR gel in the concrete mixture containing 25% ground clay brick (*Point analysis:* SiO_2= 59.60; Al_2O_3= 0.51; Fe_2O_3= 0.11; MgO= 0.32; CaO= 15.35; Na_2O= 6.08; K_2O= 13.38; SO_3= 0.30).

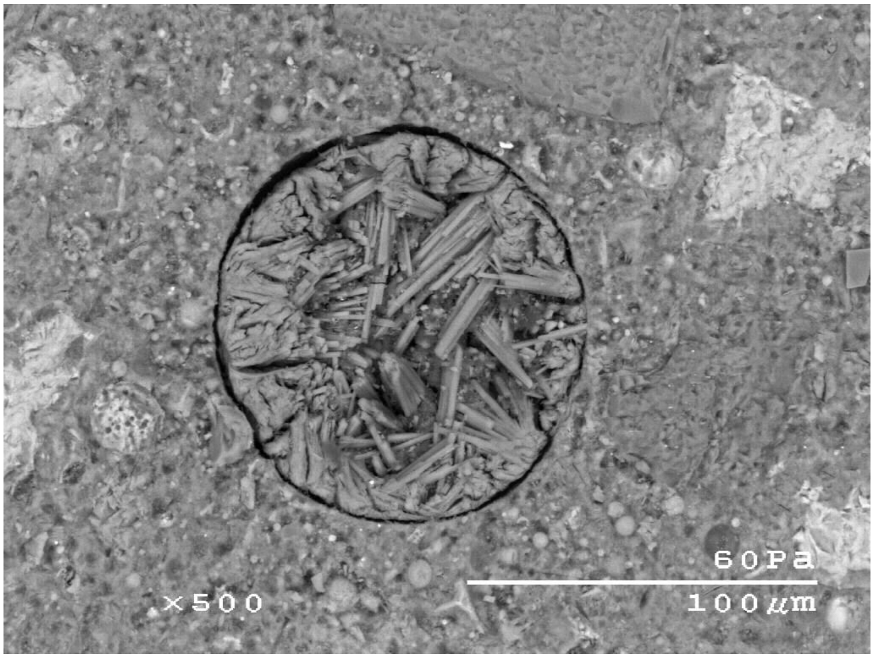


Fig. 9. Air void filled with ettringite crystals in the mixture containing 25% Class C fly ash (*Point analysis:* SiO_2= 3.42; Al_2O_3= 11.73; Fe_2O_3= 0.39; MgO= 0.13; CaO= 38.71; Na_2O= 0.11; K_2O= 0.18; SO_3= 23.29).

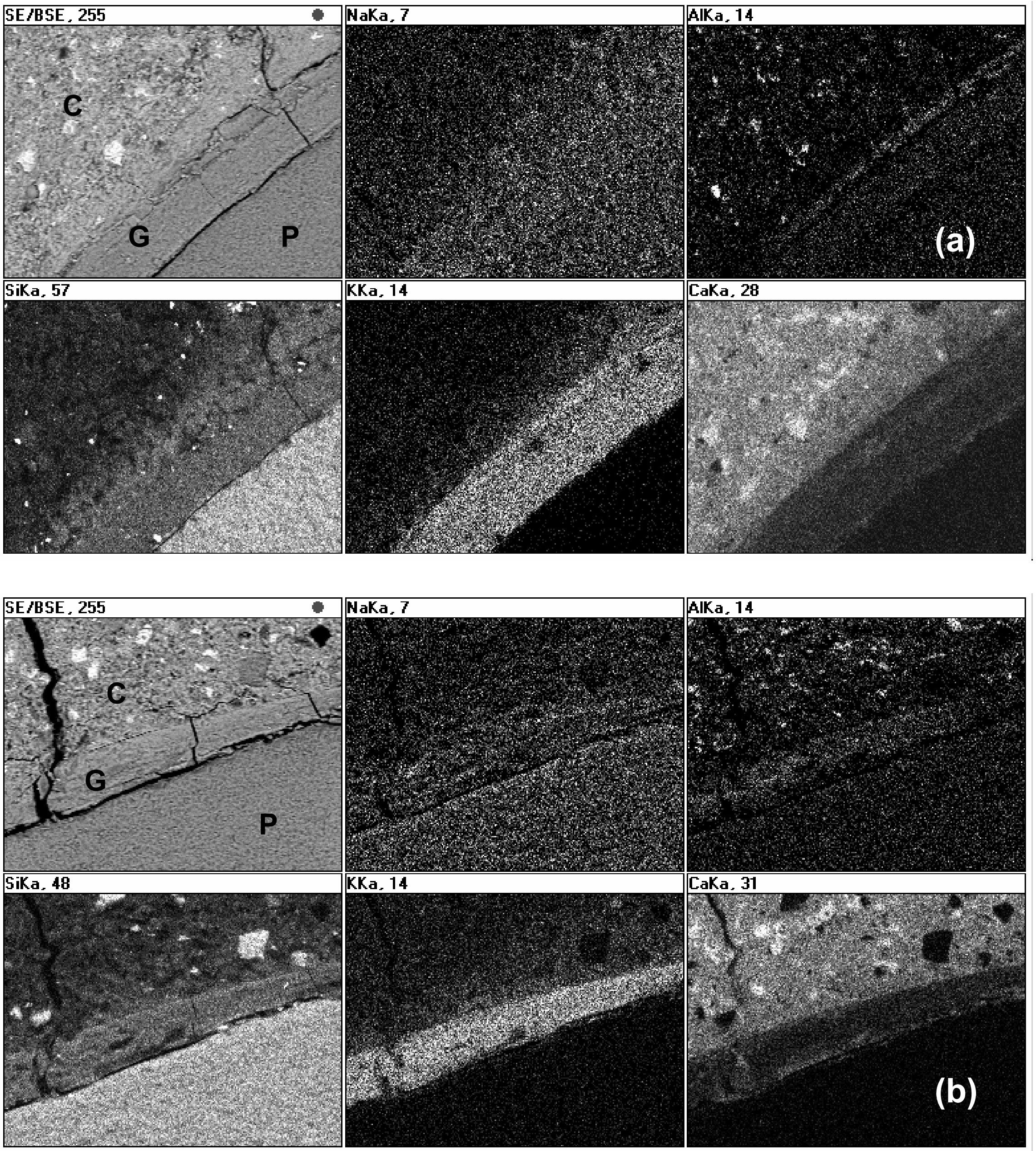


Fig. 10. X-ray mapping of ASR gel in (a) control and (b) 25% GCB samples (C=cement paste; G=ASR gel; P=Pyrex).

Chapter 6

Conclusion

This study aimed to investigate the use of ground clay brick (GCB) as a supplementary cementitious material in concrete and its effects on ASR deterioration (e.g., expansion, reduction in mechanical properties). The effect of GCB on hydration characteristics (e.g., water demand for normal consistency, time of setting, heat dissipation, $Ca(OH)_2$ content) were studied employing cementitious pastes. The effect on concrete mechanical properties (e.g., compressive, flexural, and splitting tensile strengths, and modulus of elasticity) was determined in concrete mixes where the cement was replaced by GCB up to 25%. In addition, rapid chloride ion penetration was also tested. The effectiveness of GCB in suppression of the ASR deterioration was determined in comparison to other supplementary cementitious materials, namely fly ash and volcanic tuff (natural pozzolan) employing mortar and concrete mixes. Expansion measurements, compressive and flexural strength determination were done on mortar mixes cured in NaOH solution at 80^{o}C. Furthermore, concrete mixes containing up to 25% GCB were also prepared and tested for the effectiveness of ASR mitigation. In addition to expansion measurements, compressive and flexural strengths testing, and modulus of elasticity determination were carried out. The effect of GCB on ASR gel chemistry was also studied employing scanning electron microscopy in order to provide a better understanding for the ASR suppression mechanism of GCB.

The main conclusions of the study are as follows:

- Physical and chemical properties of GCB satisfy the requirements of a pozzolanic material.
- Up to 25% GCB replacement for cement has no significant effect on the water demand and the time of setting.

- GCB replacement decreases the heat dissipation during hydration.
- GCB replacement reduces the amount of $Ca(OH)_2$ in the paste, partially due to pozzolanic action.
- Up to 25% GCB replacement, concrete slightly decreases its early age mechanical values (such as compressive, flexural and splitting tensile strengths, and modulus of elasticity). However, these values are comparable to or higher than those of the concrete without GCB at later age.
- Rapid chloride permeability (by ASTM C 1202) of concrete decreases as the amount of GCB increases.
- GCB reduces the mortar expansion due to ASR in the accelerated testing (NaOH solution at 80°C). GCB replacement (15 and 25%) effectively controls the ASR expansion of concrete.
- Flexural and modulus of elasticity are very sensitive to ASR cracking while compressive strength is relatively insensitive.
- GCB decreases the loss in flexural strength and modulus of elasticity exposed to ASR conditioning, thus reducing ASR associated degradation on concrete mechanical properties.
- The quantity of ASR gel produced is significantly less in the concrete containing GCB. The mechanism by which GCB reduces ASR expansion and deterioration can be partially explained on the basis of the modification of gel chemistry.

In summary, waste clay brick obtained from brick manufacturing reject and demolished masonry has high potential to be recycled as a supplementary cementitious material in concrete. GCB shows pozzolanic behavior similar to other conventional materials (e.g., fly ash, calcined clay, natural pozzolan). Use of GCB in concrete can effectively reduce the alkali-silica reaction deterioration in concrete.

Chapter 7

Recommendations for Future Research

The work presented here has provided concrete evidence that ground clay brick (GCB), which can be considered as a waste material, can be utilized as partial cement replacement in concrete applications. However, rurther research is needed, particularly to evaluate the economic feasibility of the material: is it worth to take this path or discard the material as waste or keep using in low value applications? These economic decisions will depend on specific cases that require accurate assessments. In terms of economic feasibility, co-grinding of brick with portland clinker can be a future research focus. From the technical point of view GCB acts like other pozzolanic materials, such as fly ash, natural pozzolan. However, more extensive work on different types of brick material in terms of origin or chemical and mineral composition would be beneficial in order to identify the suitability or the applicability of the best performing material type. The work presented here attempts to cover major issues on concrete such as strength development and ASR durability and can be used as a guide for further research, but for a more complete picture, the effect of GCB on concrete shrinkage, which is one of the main concerns regarding pozzolanic materials, may be evaluated. Freeze-thaw resistance is another area that can be studied.

Appendix A: Papers from Additional Research

Effects of crushed clay brick aggregate on mortar durability

A paper submitted to Construction and Building Materials (Elsevier)

F. Bektas[1], K. Wang[2], H. Ceylan[3]

Abstract

This paper reports an experimental study that aimed to investigate the effects of recycled clay brick, used as a part of fine aggregate, on mortar durability. The brick, in crushed form, was from a local brick manufacturer that salvages its off-standard brick products. It was used to replace 10 and 20% (by weight) of the river sand in mortar. Effects of the brick replacement on the mortar flow, compressive strength, shrinkage, freeze-thaw resistance, and alkali-silica reaction potential were investigated. The results showed that as the brick replacement level increased, the mortar flowability reduced. The 10 and 20% of the brick replacements had no negative effect on the mortar compressive strength and very limited effect on the mortar shrinkage. The freeze-thaw resistance of the mortar was improved by the brick replacement. However, the use of crushed brick as aggregate appeared not to reduce potential alkali-silica reaction. The microscopy study revealed the alkali-silica reaction product and associated cracking in the mortar. Additional study indicated that the brick aggregate used in the study had pessimum proportion, 30%, for the alkali-silica reaction expansion.

Keywords: Crushed clay brick; Aggregate; Freeze-thaw; Alkali-silica reaction; Pessimum

---

[1] Graduate student; primary researcher and author; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[2] Associate professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

[3] Assistant professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

1. Introduction

Seventy to seventy-five percent (by volume or weight) of concrete is made of aggregate which is usually derived from natural resources. As aggregate sources become depleted in an increasing rate, the industry tries to implement alternate resources to meet the demand. Recycling aggregate from demolition rubble is one of the prudent methods: it is not only economically viable, particularly, in urban areas, but also targets an environmental friendly approach.

Annual construction and demolition waste production in the developed countries is at the levels of hundred million tons. For the United States, the figure was estimated as 136 million tons by the Environmental Protection Agency [1]. Similarly, the construction and demolition waste in the first 15 countries of the European Union was reported to be 180 million tons per year [2]. Significant portion of this demolition waste is masonry rubble consisting of clay bricks. In addition, brick and tile manufacturing industry produces a large number of rejected fired products due to being off-standard: these unites may be broken, distorted, underburnt, or overburnt. This material is sold for landscaping purposes, if economically feasible, but usually dumped into sites around the brick factories or recycled in concrete as aggregate where natural rock deposits are scarce [3, 4].

In the 20th century, numerous research studies had been done in an effort to evaluate the potential of crushed brick as concrete aggregate. Systematic investigations on the practice of crushed brick aggregate have been carried out since 1928, particularly, in 1940s and 1950s. After the World War II, in Germany, it was necessitated to remove the rubble left from the war era and to provide adequate aggregate supply for the new construction. Rubble recycling plants were built. The benefit would be two-fold: removal of the rubble and supply of aggregate for the new concrete. These plants were reported to produce around 11.5 million m^3 crushed brick aggregate by the end of 1955 with which 175,000 dwelling units were built [5]. After that era, research on brick aggregate has been continued to date. It has been proved that using crushed brick aggregate, it is quite possible to produce concrete with adequate properties for a wide range of applications [3-11].

Research on crushed brick has generally been concentrated on its effects on the mechanical performance when brick is used as coarse aggregate in concrete. On the other hand, research concerning durability is limited. The goal of the study reported in this paper was to evaluate the effects of crushed clay brick fine aggregate on durability employing mortar specimens. The mortar aggregate was partially replaced by crushed brick at levels of 10% and 20% by weight, and the effects of crushed brick addition were investigated on compressive strength, shrinkage, freeze-thaw, and alkali-silica reaction (ASR). A special emphasis was given to the ASR investigation since there is no reported case involving crushed clay brick aggregate within the knowledge of the authors.

2. Materials

Portland cement (ASTM Type I) with a Blaine fineness of 368m^2/kg was used in this experimental study. The chemical composition is given in Table 1. River sand and crushed clay brick (referred as brick or crushed brick in this paper) were employed as mortar aggregate. The brick was obtained from a local brick manufacturer who was selling it for landscaping applications. It was further reduced in size and the part passing No.4 (4.75mm) sieve was used. The gradations are shown in Fig. 1. In the ASR tests, manufactured limestone sand, which was utilized as non-reactive component, and crushed brick were graded in accordance with the ASTM C1260 requirement and blended in desired proportions.

3. Experimental methods

The crushed brick was introduced as weight percentage of the mortar aggregate. Essentially, two levels of addition, 10 and 20%, were investigated. However, higher percentages were also tested depending on the specific purpose such as 30% for the flowability, and 5, 30, 50 and 100% for the ASR activity. The initial mixtures were B0 (control), B10 and B20. The number at the end denotes the percentage of the crushed brick in the mixture. ASTM C305 mixing procedure was followed to prepare the specimens. Cube specimens (50mm) were cast to test compressive strength. Mortar bars of 25×25×285mm^3 were used for shrinkage, freeze-thaw, and ASR measuremens. A fixed water-cement ratio of 0.50 was utilized for all the mixture proportions. Cement-aggregate ratio was 1:2.75 except for the alkali-silica test being 1:2.25.

The compressive strength was tested up to 1 year in accordance with ASTM C109. The same mixtures were used to determine the flow according to ASTM C1437. Additionally, a 30% brick addition was tested for the flowability. For the shrinkage testing, the mortar bars were stored in the normal lab environment: the temperature and the relative humidity were in the ranges of 19-25 ^{o}C and 35-55%, respectively. The bars were allowed for free shrinkage and the length changes were recorded up to 56 days. For the freeze-thaw testing the mortar bars were exposed to ASTM C666 Procedure A conditions: the specimens were kept in fully saturated condition with temperature cycling between -17.8 and 4.4^{o}C, each cycle being 4 hours. The ASR testing was carried out according to the accelerated mortar bar test, ASTM C1260. The mortar bars were demolded after 24 hours of moist curing and cured for another 24 hours in water bath at 80^{o}C. The bars, then, were stored in 1N sodium hydroxide (NaOH) solution at 80^{o}C. The vertical length change was reported as the measure of expansion. Representative cross sections (25×25mm^2) were cut using a low speed saw and dried at 50^{o}C. The sections were, then, epoxy impregnated under vacuum and the surface was polished for microscopy examination. The micro study was carried out by employing a variable pressure scanning electron microscope (SEM) equipped with energy dispersive x-ray spectrometer (EDS) for chemical analysis.

4. Results and discussions

4.1 Flowability

Fig. 2 shows the mortar flow results together with the best fit. It is apparent that the flowability, which is a measure of mortar workability, decreases as the crushed brick content increases. Compared to the control sample, the reduction was 11, 23, and 32% for the addition of 10, 20, and 30% brick aggregate, respectively. The trend is almost linear as 10% reduction occurs for each additional 10% brick aggregate. It is obvious that the high water absorption capacity of the brick significantly affects the mortar flow. Water absorption of the brick used in this study was found as 7.00% according to ASTM C128. Brick is a porous construction material whose porosity may be as high as 40% depending on the raw material and manufacturing process, hence, aggregate produced from brick has high water absorption. Together with the angular shape the high water absorption results in mixing problems in concrete production. Pre-soaking or adding extra water in account of absorption has been recommended [3,6]. On the other hand, even 30% brick incorporated mixture had sufficient workability and good consolidation with the given mixture proportions in this study.

4.2 Compressive strength

Fig. 3 demonstrates the compressive strength results obtained from the mortar cubes. Each data point represents the average of three cubes. The value for B20 at 6 months is not reported due to insufficient data. For all the mixtures, there is a gradual increase with the aging. When the mixtures are compared, it is seen that the values are all close to each other which confirms the brick aggregate does not cause strength reduction with the levels used. Khatib [12] utilized fine crushed brick aggregate in concrete and found that 25% brick replacement has the same 90-day strength as the control does. The 28-day ratio was only 0.84: the strength gain between 28 and 90 days was attributed to the pozzolanic action provided by the very fine portion of the brick. In a recent study, Debieb and Kenai [13] reported 5-10% strength reduction for 25% fine brick aggregate replacement; th reduction is 30% for full fine aggregate replacement. The studies usually involve the use of brick replacing coarse aggregate in varying proportions. Depending on the strength of the parent brick and on the other concrete parameters, a wide range of compressive strength can be obtained: De Brito et al. [10] reported 13MPa 28-day strength when the coarse aggregate was replaced with crushed brick whereas Khalaf and De Venny [9] achieved high strength concrete - 67MPa at 28 days.

4.3 Shrinkage

Fig. 4 shows the results of the shrinkage measurements obtained from the mortar bars: each data point represents the average of three bars. The mortar containing 10% crushed brick showed the highest shrinkage. On the other hand, 20% crushed brick addition resulted in the least shrinkage value. Khatib [12] also reported a decrease in the shrinkage as the content of crushed brick increases. The same author's study also showed an expansion increase with the crushed brick content when the specimens were stored in water and the expansion was discussed to occur due to the pozzolanic gel which imbibes water: a higher expansion is associated with lower shrinkage with the exception of the control mix. Moreover, Mansur et al. [3] observed lower shrinkage when crushed clay brick aggregate replaced the same volume of crushed granite. The brick particles may be acting as a self-curing agent: it keeps the initially absorbed mixing water in its pores for longer periods and releases it slowly as aged. Therefore, drying shrinkage is delayed since the hydration continues due to the presence of internal moisture [14].

4.4 Freeze-thaw

Fig. 5 presents the expansion curves of the mortar bars subjected to freeze-thaw cycles. It is worth noting that one of the bars from the control mix had broken after few cycles due to cracking and another one around 120 cycles. B10 and B20 data points are the average of three bars. In order to attain adequate strength development, the mortar bars were moist cured for 28 days before exposed to the freeze-thaw cycles. The plot clearly demonstrates that B20 mixture achieved the best performance. As the quantity of the crushed brick increases the expansion caused by the freeze-thaw action decreases. After

180 cycles, CB20 showed an average expansion of 0.05% whereas the control mix expanded 0.53%. Visual inspection also confirms that the surface scaling of the control bars were greater as compared to the brick incorporated ones. The idea behind the inclusion of air bubbles to prevent freeze-thaw associated cracking is to relieve the pressure caused by ice formation and water flow, and to cut the water flow paths; in other words, closely distributed air void structure provides space for expansive mechanisms. Similarly, the highly porous nature of crushed brick might provide air entraining action and reduce the freeze-thaw expansion. This concept is successfully applied by Litvan and Serada [15]. Freez-thaw resistance of hardened concrete was increased by the incorporation of porous brick particles which are 0.5 ± 0.08 mm in size and has 36 percent total porosity: the specimens containing 16% (by weight) brick endured 1260 freeze-thaw cycles without damage.

4.4 Alkali-silica reaction

The results of ASTM C1260 test is plotted in Fig. 6. Manufactured limestone sand was employed as the non-reactive component. It was proved to be non-reactive (B0 in Fig. 6). ASTM 1260 specifies an aggregate as potentially alkali reactive if the expansion exceeds 0.20% at the end of 14 days. The results showed that the 10 and 20% brick addition resulted in deleterious expansion. The expansions were 0.30 and 0.35% for B10 and B20, respectively. The cracking on the bars are visible to bare eye. Clay brick is produced from siliceous earth, thus, the final product being siliceous would not be surprising to show ASR activity. Furthermore, vitrified clay pipe is known to be resistant to most chemicals except hydrofluoric acid and caustic soda (NaOH) [16].

The SEM study demonstrated the internal disruption of the mortars with no doubt. The expansive force cracked the cement matrix and the non-reactive limestone particles as well. Fig. 7 shows the reactive brick particles and internal cracking. The x-ray dot map shown in Fig. 8 proves the siliceous brick aggregate acts as attraction sites for the sodium. X-ray mapping is a qualitative method providing information about the concentration of the element in a gray scale: lighter shades showing the abundance of the element analyzed. Fig. 8b clearly demonstrates the border line between the brick particle and the calcium-rich cement paste. The brick is mainly composed of silica and aluminum (Figs. 8c and 8d) and some iron as well (Fig 8e). Fig. 8f demonstrates that there is a concentration increase of sodium on the area occupied by the brick particle. The reaction product, well-known alkali-silica gel, was observed in the samples under investigation. It was evident that the gel moves along the cracks to find out escape boundaries filling the voids and lining the cracks (Fig. 7). Moreover, Fig. 9 clearly identifies the gel in the cracks of a limestone particle. Fig. 9f shows the traces of the potassium in the gel as well. The source was attributed to the cement. The siliceous brick particle was found to be sensitive to alkali considering the potassium concentration is very low compared to the sodium in the environment.

The authors' experience has showed that the highly reactive materials tend to have pessimum content (or proportion) at which reactive aggregate leads to the maximum expansion. In order to determine the pessimum proportion, additional study was performed: mortar bars containing 5, 30, 50, and 100% of the brick aggregate were cast and subjected to ASR testing. The results (Fig. 10) showed that the crushed brick aggregate used in the study has pessimum content at around 30%. To the authors' knowledge there is no ASR case reported with the involvement of clay brick aggregate. In practice brick is generally replaced as coarse aggregate. Utilization higher than the pessimum proportion or high porosity of brick entrapping the reaction gel may be possible explanation for no deleterious reaction reported, or simply the conditions have not been favorable. However, this phenomenon should be further investigated in concrete and ASR remedies should be considered if brick aggregate is intended to be utilized.

5. Summary

Crushed clay brick obtained from a local brick manufacturer was introduced into the mortar as a percentage of the total aggregate. The experimental study showed that the brick negatively affected the mortar flow. The brick aggregate, when added in 10 and 20%, had no negative effect on the compressive strength. The 20% crushed brick reduced the mortar bar shrinkage whereas 10% increased as compared to the control mix. Furthermore, the crushed brick decreased the freeze-thaw associated expansion in proportion to the addition amount. However, the brick used in the study was found to be highly alkali-reactive with pessimum content at 30%. ASR susceptibility of the clay brick aggregate merits further study and the extent of its effects on concrete should be evaluated in detail in order to prevent further complications with the use of crushed clay brick aggregate in concrete.

References

[1] http://www.epa.gov/epaoswer/non-hw/debris-new/basic.htm

[2] Pladerer C. Initiatives for a sustainable plastic C & D waste management in Europe: The case of Austria. In: European Workshop – Towards Sustainable Plastic Construction and Demolition Waste Management in Europe, Brussels, Belgium, 2006.

[3] Mansur MA, Wee TH, Cheran LS. Crushed brick as coarse aggregate for concrete. ACI Materials Journal 1999; 96 (4): 478-484.

[4] Mazumder AR, Kabir A, Yazdani N. Performance of overburnt distorted bricks as aggregates in pavement works.

[5] Khalaf FM, DeVenny AS. Recycling of demolished masonry rubble as coarse aggregate in concrete: review. Journal of Materials in Civil Engineering 2004; 16(4): 331-340.

[6] Khaloo AR. Properties of concrete using crushed clinker brick as coarse aggregate. ACI Materials Journal 1994; 91(2): 401-407.

[7] Zakaria M, Cabrera JG. Performance and durability of concrete made with demolition waste and artificial fly ash-clay aggregates. Waste Management 1996; 17(1-3): 151-158.

[8] Padmini AK, Ramamurthy K, Mathews MS. Behavior of concrete with low-strength bricks as lightweight coarse aggregate. Magazine of Concrete Research 2001; 53(6): 367-375.

[9] Khalaf FM, DeVenny AS. Performance of brick aggregate concrete at high temperatures. Journal of Materials in Civil Engineering 2004; 16(6): 456-464.

[10] De Brito J, Pereira AS, Correia JR. Mechanical behavior of non-structural concrete made with recycled aggregate. Cement and Concrete Composites 2005; 27(4): 429-433.

[11] Poon CS, Chan D. Paving blocks with recycled concrete aggregate and crushed clay brick. Construction and Building Materials 2006; 20 (8): 569-577.

[12] Khatib JM. Properties of concrete incorporating fine recycled aggregate. Cement and Concrete Research 2005; 35(4): 763-769.

[13] Debieb F, Kenai S. The use of coarse and fine crushed brick as aggregate in concrete. Construction and Building Materials; *article in press*.

[14] Hansen IC. Recycling of demolished concrete and masonry. RILEM Report of Technical Committee 37-DRC (Demolition and Recycling of Concrete), RILEM Report 6. London, UK: E&FN Spon, 1992.

[15] Litvan GG, Sereda PJ. Particulate admixture for enhanced freeze-thaw resistance of concrete. Cement and Concrete Research 1977; 8(1): 53-60.

[16] The advantages of clay in today’s industrial sewer pipe applications. http://www.loganclaypipe.com/whitepaper.htm

Table 1. Chemical composition of the portland cement

Oxide	%
CaO	64.61
SiO_2	20.58
Al_2O_3	5.38
Fe_2O_3	2.14
MgO	2.08
K_2O	0.46
Na_2O	0.26
SO_3	3.01
Free Lime	1.00

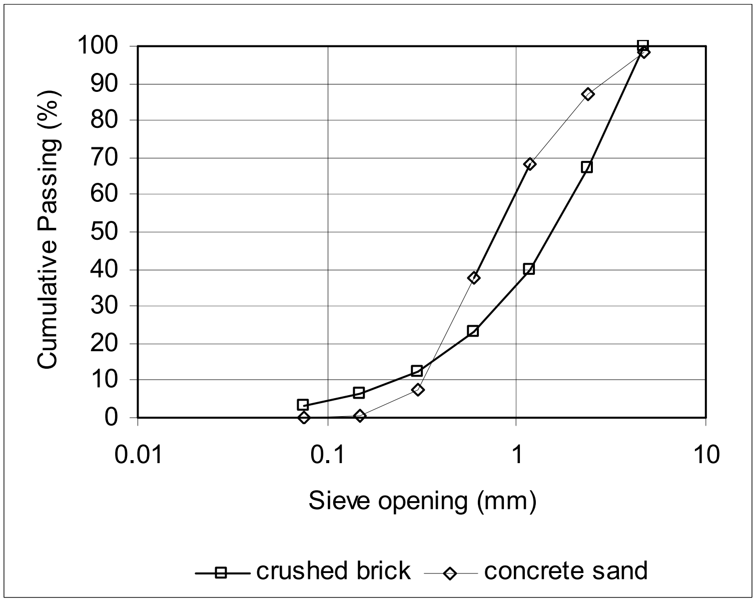


Fig. 1. Gradation of the aggregates used in the mortar mixtures.

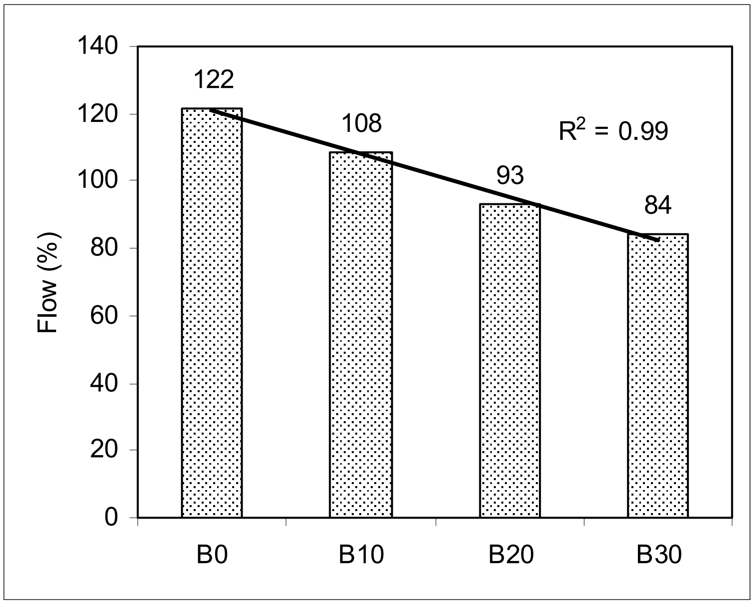


Fig. 2. Effect of the crushed brick on the workability.

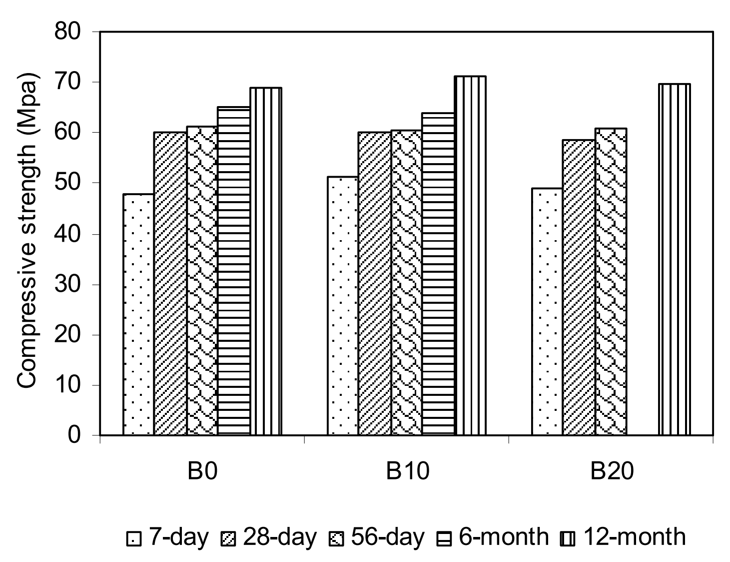


Fig. 3. Compressive strength of the mortar cubes.

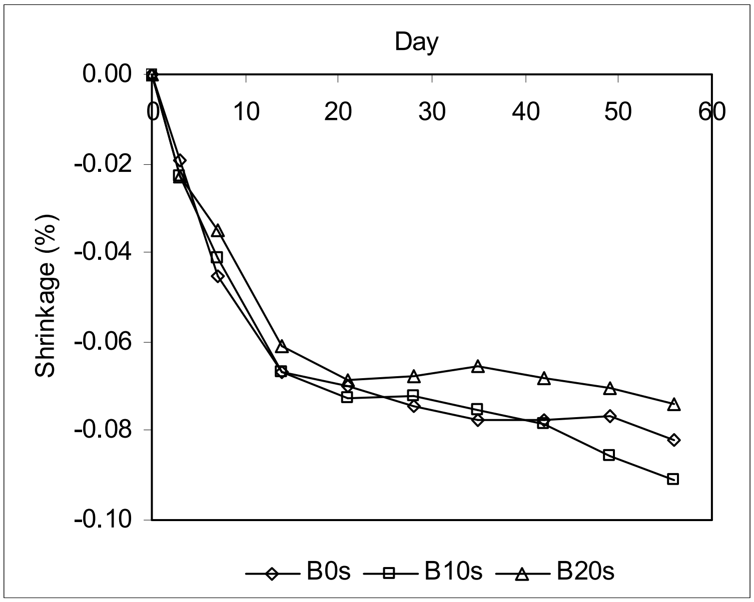


Fig. 4. Length change of the mortar bars due to shrinkage.

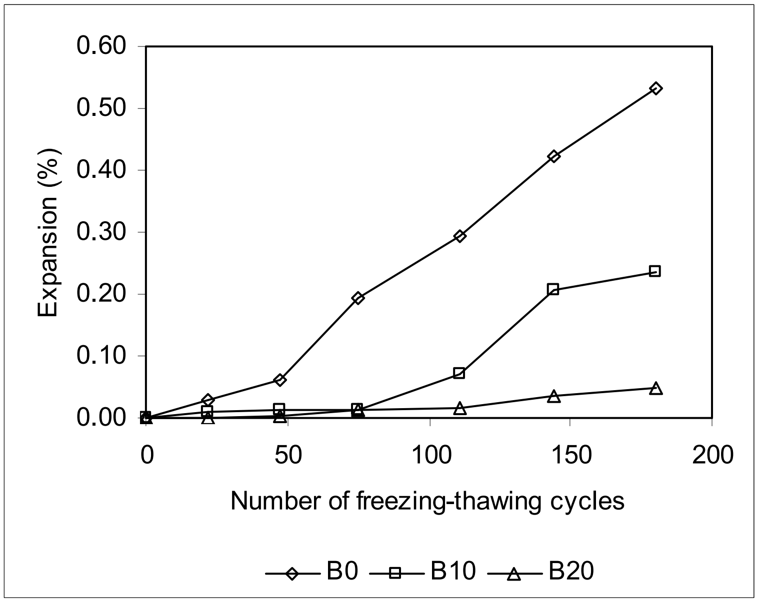


Fig. 5. Expansion of the mortar bars due to freeze-thaw action.

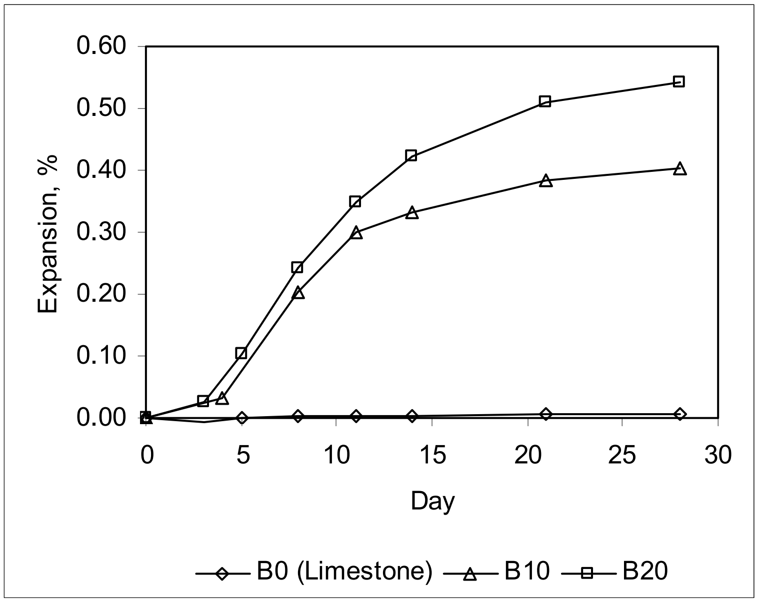


Fig. 6. Expansion of the mortar bars tested according to ASTM C1260.

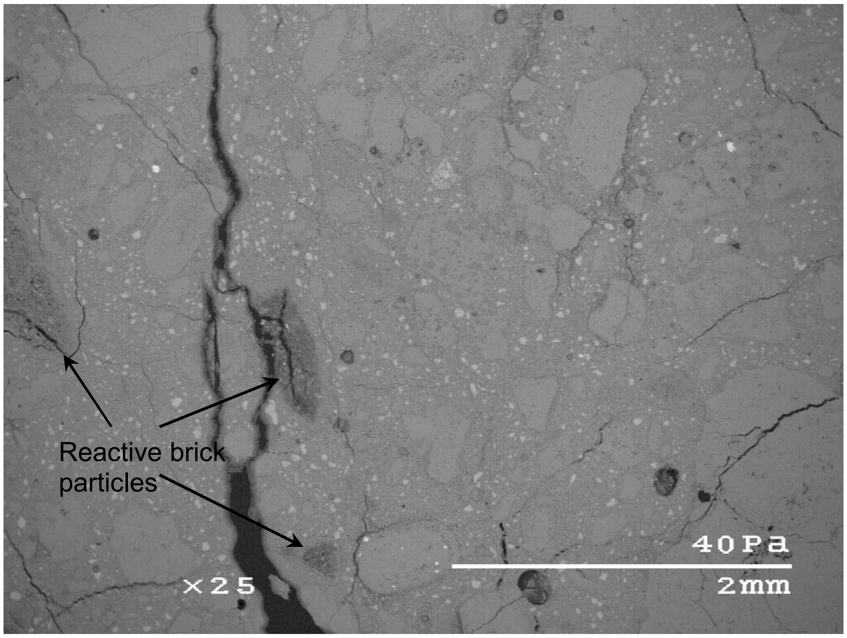


Fig. 7. The internal mortar cracking due to ASR (B20 sample at 28 days).

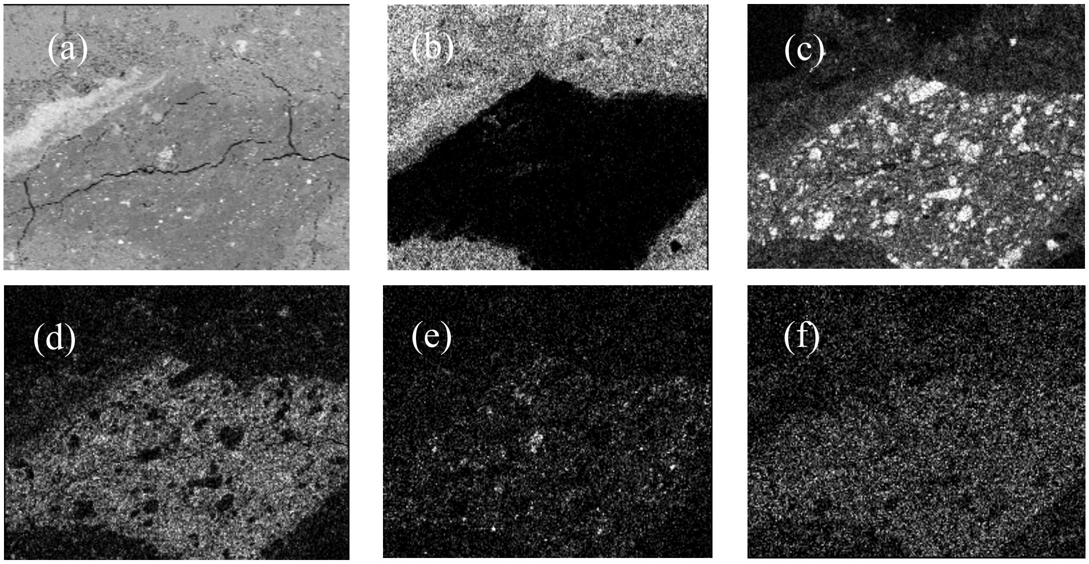


Fig. 8. X-ray dot maps of the reactive brick particle – B10 sample *(a)* backscattered image; *(b)* Ca; *(c)* Si; *(d)* Al; *(e)* Fe; *(f)* Na.

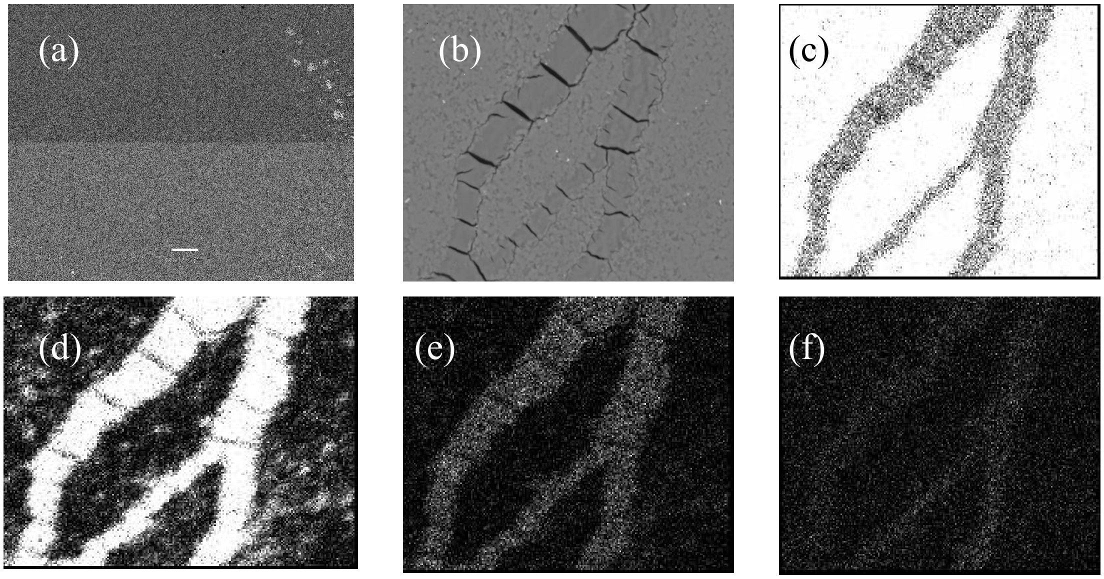


Fig. 9. X-ray dot maps of the ASR gel in the crack – B20 sample *(a)* backscattered image; *(b)* area marked on the low magnification image; *(c)* Ca; *(d)* Si; *(e)* Na; *(f)* K.

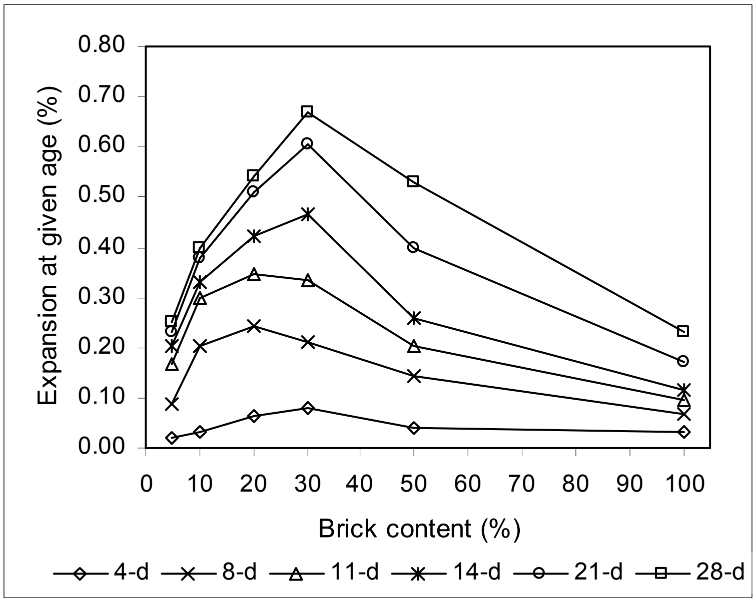


Fig. 10. The crushed brick aggregate showing pessimum behavior.

Effect of Portland Cement Fineness on ASTM C 1260 Expansion

A paper submitted to Journal of Testing and Evaluation (ASTM)

F. Bektas[1], K. Wang[2], H. Ceylan[3]

ABSTRACT

The present study is aimed at investigating the effect of portland cement fineness on the results of ASTM C1260 tests - *Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-bar Method)*. The effects of clinker alkali content, aggregate reactivity, and sodium hydroxide (NaOH) solution concentration on the mortar expansion test results were also studied. In this study, high and low alkali portland cement clinkers were selected and ground with gypsum in a laboratory ball mill to three fineness levels - 300, 400 and 500 m^2/kg (Blaine). Moderately- and highly-reactive aggregates were tested with these cements according to ASTM C 1260. In addition to the standard 1 *N* NaOH solution, a 0.5 *N* NaOH soak solution was also used. The results show that mortar-bar expansion was promoted with increased cement fineness regardless of clinker alkali, aggregate reactivity or soak solution normality. Clinker alkali had no or little effect on moderately reactive aggregate; whereas it had considerable effect on highly reactive aggregate. Highly reactive aggregate tended to be more sensitive to cement fineness and alkalinity. Although cement fineness and clinker alkali content did not affect the decision on potential reactivity of the aggregates used in this study, they might change the decision for the aggregates near the boundary between reactive and non-reactive aggregates.

---

[1] Graduate student; primary researcher and author; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.
[2] Associate professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.
[3] Assistant professor; Civil, Construction, and Environmental Engineering Department, Iowa State University, Ames, IA 50011.

KEYWORDS

ASTM C 1260, alkali-silica reaction (ASR), ball mill, Blaine, expansion, portland cement fineness

Introduction

Since its initial discovery by Stanton in the early 1940s, alkali-silica reaction (ASR) has been one of the major durability concerns of the concrete industry. ASR is the chemical interaction of concrete alkalis and reactive siliceous aggregate. The reaction results in a gel-like product which is capable of swelling in presence of water. The swelling pressure is often sufficient to cause concrete cracking.

Detection of reactive aggregate prior to use in concrete is crucial in order to take actions preventing the deleterious reaction. Numerous test methods have been proposed after decades of research [1]. Since ASR is a slow process, harmful chemical and environmental effects on concrete may take years to exhibit. Therefore, almost all ASR tests involve the acceleration of the reaction by aggravating one or more of the following influential factors:

- Alkali concentration
- Temperature
- Humidity

ASTM C 1260 – Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-Bar Method) [2], or more generically the accelerated mortar-bar test (AMBT), has been intensively used all around the world under different codes. Table 1 summarizes the AMBTs used in different parts of the world [2–6]. The AMBT is known to be based on the South African National Building and Research Institute (NBRI) method which was proposed by Oberholster and Davies [7]. The test has been become very popular since it is relatively quick and easy to perform. The method requires periodic length measurements of 25 by 25 by 285 mm mortar-bars which are immersed in 1 *N* NaOH solution at 80°C. The length change, or expansion, after 14 days immersion (16 days since casting) is taken as the indication of potential reactivity. ASTM C 1260 considers expansion of > 0.20% as reactive and < 0.10 as innocuous; the values fall in 0.10 % to 0.20 % range is inconclusive and requires additional testing but mostly referred to potential reactivity (See Table 1 for the reactivity criteria in other AMBTs). Although being widely accepted and used, the AMBT draws criticism as having over-aggressive conditioning: some innocuous aggregate with proven field performance are found to be reactive. Therefore, it is common agreement that an aggregate should not be assessed as reactive solely based on ASTM C 1260 results [8].

It is well-accepted that the hydration rate of cement increases with cement fineness. This is almost true for all chemical reactions that occur between a solid and a liquid or gas, which is promoted with the increasing surface area of the solid. Similarly, when a finer cement is used, the increased rate of alkali release from the cement may aggravate the deleterious ASR in the concrete. Higginson [9] observed an increasing trend in the expansion of concrete made with reactive aggregate and high-alkali cement as the cement fineness goes from 220 m^2/kg to 360 m^2/kg Blaine. ASTM C 1260 requires the use of portland cement meeting ASTM C 150. The Specification C 150 states a lower limit of 280 m^2/kg of fineness measured with the Blaine apparatus (ASTM C 204). However, with regard to the fineness, a wide variety of material can be used. According to a survey [10] on the portland cements characteristics produced in the North America in the late 1990s, the ranges of fineness are 310 to 497 m^2/kg and 319 to 672 m^2/kg for Type I and III cements, respectively. This wide range permits the cements produced from the same clinker with fineness at the far ends of the scale, may possess different properties to be used in the AMBT. Berra et al. [11], in their study testing natural reactive sands with ASTM C 1260, concluded that the native alkali content and the specific surface area of the portland cement are significant factors in affecting mortar-bar expansions.

Although there have been studies concerning the effect of alkali content on ASTM C 1260 expansion, the effect of fineness has apparently never been given further attention. In this study, a detailed and controlled approach was undertaken to investigate the effect of portland cement fineness on ASTM C 1260 expansion in conjunction with other influential factors, namely alkali content of clinker, aggregate reactivity, and immersion solution concentration.

Experimental Work

Materials

Two types of clinker (high alkali and low alkali) were used in the study. Table 2 gives the chemical composition of portland cements produced from these clinkers. Two different reactive fine aggregates, moderately and highly reactive, were used in the study. Their origins are Nebraska, USA (denoted as "NE") and New Mexico, USA (denoted as "NM"). Petrography with regard to alkali reactivity showed that NE contains metamorphosed granite with strained quartz; there are also minor amounts (1.0-1.5 %) of volcanic glass and cryptocrystalline chert. On the other hand, NM aggregate contains significant amounts of volcanic glass (4%) and chert (2 %), in terms of alkali-reactivity. In addition, andesite, granite and recrystallized quartz particles are also present in the NM sand. The aggregates were sieved and recombined in accordance with the grading requirements of ASTM C 1260.

Production of Portland Cements

A custom-designed laboratory-type ball mill was used for the production of the portland cements. The ball mill has dimensions of 51 cm in diameter and 28 cm in length. It provides 30 RPM when loaded. Raw materials, clinker and gypsum, were crushed using a jaw crusher and passed through 2.36 mm (ASTM No. 8) sieve before final grinding. A dry grinding process was utilized. Both materials were dried in order to remove moisture which might lead to sticking of particles to balls and mill surface. Steel balls were used as grinding media. Ball charge was selected as 90 kg: equal weights of 37.5 mm and 25.0 mm spherical balls. A batch of 7.0 kg material (96.7 % clinker and 4.3 % gypsum by weight) was fed into the mill.

The aim of the laboratory ball mill grinding was to produce portland cement with controlled levels of fineness. To achieve this goal, the grinding curve (fineness versus grinding time) was first developed for each clinker. The cement fineness was determined in accordance with the Blaine method, ASTM C 204 – Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus [12]. Based on the grinding curve, portland cements with desired fineness levels were produced at a given grinding time and the actual fineness of the manufactured cement was also measured.

ASR Testing – ASTM C 1260

Mortar-bars were prepared using the available reactive aggregates and the laboratory produced cements. The bars were produced and tested in accordance with ASTM C 1260:

- The mortar was mixed in accordance with ASTM C 305 – Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency [13].
- The bars were cast in steel molds which were covered with removable tapes and plastic wraps as release agent.
- The bars were demolded after 24 h and cured in water at 80°C for another 24 h.

- Initial reading was taken after demolding and zero reading was taken at the end of 24-hour hot water curing. Then the bars were transferred into 1 N NaOH solution. A 0.5 *N* solution was also used for conditioning.
- The mortar-bars were stored in air- and water-tight polypropylene containers in a laboratory type oven.
- The length change of the bars was recorded periodically up to 28 days instead of 14 days recommended by the standard.

Testing Parameters

The experimental work includes the following testing parameters in order to investigate the effect of portland cement fineness on ASTM C 1260 expansion:

- 3 fineness levels: 300, 400, 500 m^2/kg Blaine
- 2 clinkers: low alkali (~0.4% Na_2O_{eq}) and high alkali (~0.9% Na_2O_{eq})
- 2 reactive aggregates: moderate reactivity (NE) and high reactivity (NM)
- 2 different levels of NaOH solution normality: 1.0 *N* and 0.5 *N*

Results

Grinding Curve

Grinding curves of the clinkers are given in Fig. 1 including all the trial batches and the final products. Three fineness levels of cement were targeted in the study – 300, 400 and 500 m^2/kg termed as H3, H4 and H5 or L3, L4 and L5 where H indicates high alkali clinker and L represents low alkali clinker, and the preceding number represents the fineness. Desired fineness levels were achieved in a range of ±20 m^2/kg. The exact values are tabulated in Table 2. The low alkali clinker was harder than the higher alkali clinker so that a longer grinding time was required to achieve the same fineness.

ASTM C 1260 Expansion

At the end of testing period (extended to 28 days), mortar-bars made with both NE and NM aggregate showed surface cracking regardless of the cement and solution concentration. The degree of cracking, in terms of surface area and crack width, was higher for the mortars made with the highly reactive NM aggregate than that of the mortars made with moderately reactive NE aggregate. Some of the mortar-bars made with NM aggregate even exhibited warping, which tends to be associated with high expansion.

The ASR expansions of the mortar-bars cast with the laboratory produced cements are given in Figs. 2 to 5. Each data point represents the average of four bars. The figures clearly indicate that there is an increase in the expansion as the fineness increases: the trend is valid regardless of clinker alkali, aggregate reactivity, and solution normality. However, the effect is more significant for the highly reactive NM aggregate.

Fig. 2 gives the expansion curves of the moderately reactive NE aggregate tested in different solution concentrations with different fineness levels of the higher alkali clinker; and Fig. 3 provides the same data for highly reactive NM aggregate. The effect of solution normality is worth mentioning: the ultimate expansions are comparable for the moderately reactive aggregate and even higher for the highly reactive NM aggregate. However, the incubation period of the reaction is extended in the solution with low normality. The behavior is identical for the mortar-bars cast with the low alkali cements (Figs. 4 and 5).

Discussion

Fig. 6 summarizes the effect of parameters on the 14-day expansion. The effect of fineness is clearly observed in the figure, particularly, for the highly reactive aggregate. The expansion is almost linearly correlated with the cement fineness. Fournier and Berube [14] also observed higher expansion in the accelerated mortar-bar method with the highest Blaine values. In their study with different clinkers, Berra et al. [11] reported exacerbating effect of fineness on the ASTM C 1260 expansion. Their results also indicate that the fineness effect is limited when using moderately reactive aggregate, which is in good agreement with the results of this study.

Fig. 6 further indicates that the effect of cement alkali content on the expansion is negligible for the moderately reactive aggregate; however, it is significant for the highly reactive aggregate: for the same fineness, higher alkali cement produced higher expansion. Although it is commonly accepted that the initial alkali level of the mixture has no significant effect on expansion because specimens are immersed in 1 *N* NaOH solution, contradictory data is also available in the literature. Hooton and Rogers [15]

used two Portland cements with alkali contents of 0.40 % to 0.92 % and found an 8.2 % increase in expansion from low to high alkali cement and reported alkali content has a minor effect on expansion. On the other hand, Owsiak [16] reported parallel findings with significant difference: mixtures with 4% reactive opal produced 14-day expansions of 0.3, 0.6 and 0.7% with an initial mixture Na_2O_{eq} of 0.35, 0.70 and 1.10% by cement weight. Similarly, Berra et al. [11] claimed that the alkali content of cement has significant effect on ASTM C 1260 expansion and explained that there is a threshold level for alkali concentration in the pore solution within the mortar, above which the expansivity of the aggregate is promoted and sustained. This threshold exists even for the mortar-bars immersed in NaOH solution and is affected by the fineness and alkali content of the portland cement, and also by the permeability of the mortars.

The results of this study showed that the fineness and alkalinity of cements did not affect the classification of the aggregates tested as deleterious or innocuous. However, different fineness and different alkali content cements are capable of producing different amount of expansion within a given limit for a given aggregate (Figs. 7 and 8). With regard to 14-day expansion, for the moderately reactive aggregate, the band is narrow, 0.12 – 0.14 %; however, for the highly reactive aggregate the band is considerably wide, 0.55 – 0.73 %. This wide range may lead to false decisions for certain aggregates especially when the difference between reactive and non-reactive is defined as a line. To minimize variation in expansion arising from the investigated cement characteristics, namely cement fineness and alkali, limits could be adopted in the ASTM accelerated mortar-bar method. CSA specifies cement alkali content as 1.0 ± 0.1 % and RILEM recommends the use of high alkali reference cement. As for the fineness there is no clear specification in the current practices. RILEM committee working on AMBT proposed to use >1.0 % Na_2O_{eq} cement with a minimum Blaine fineness of 450 m^2/kg [17] but did not pursue the recommendation on fineness in the later studies. The inconclusive buffer zone in ASTM is useful. As a further step a two step criterion, defining two reactivity limits at two different ages might be useful.

The cement fineness also has an effect on expansion for the mortar-bars immersed in low solution concentration, 0.5 *N*. However, it is worth to note that the comparable expansion values observed in the low normality solution is not in agreement with the available literature [18]. On the other hand, ASR expansion is a result of a complex mechanism which is dependent on sensitive chemical equilibriums that have not been clearly explained. One case may not match another. For instance, Davies and Oberholster [7] proposed 1 *N* solution concentration as the optimum since it produced higher expansion than 1.5 *N*. However, Fournier and Berube [14] reported higher expansions for 1.5 *N*. Furthermore, ASTM C 1260 measures expansion depending, solely, on linear length change; however, in reality expansion is multidirectional. As mentioned earlier some mortar-bars in the study experienced warping, which may affect the measurements expressed in one direction. ASTM C 227, which also involves the length measurements of mortar-bars for ASR, recommends the documentation of warping. The same record might be helpful if included in ASTM C 1260.

Conclusions

Portand cements with different fineness levels, namely 300, 400 and 500 m^2/kg, were produced in the laboratory using high and low alkali clinker. These cements were used to test moderately and highly alkali reactive aggregates in 0.5 *N* and 1 *N* NaOH solution following the ASTM C 1260 test procedure. The following conclusions are drawn based on the test results:

- As the cement fineness increased, the ASTM C 1260 expansion also increased regardless of aggregate reactivity, clinker alkali content, and NaOH solution concentration. The effect is more significant for the highly reactive aggregate.
- Cement alkali content did not significantly affect the expansion of the mortar made with the moderately reactive aggregate, but tended to have significant effect on the expansion of the mortar made with the highly reactive aggregate.
- Cement fineness and alkali content did not affect the decision on potential reactivity of the aggregates used in this study; however, it evidences that they might change the decision for the aggregate that falls near a limit line. Specification of fineness and/or alkali content of cement is useful to further improve the reliability and reproducibility of ASTM C 1260.

Acknowledgement

The study was funded by the 2005 Portland Cement Association (PCA) Education Foundation Research Fellowship Program. The authors would like to thank to Steven

Kosmatka (PCA), Beatrix Kerkhoff (PCA) and Paul Tennis (PCA) for their support of this research. The gratitude is also extended to the North American cement manufacturers which provided clinker for the study.

References

[1] Hobbs, D.W., *Alkali-Silica Reaction in Concrete*, Thomas Telford Ltd., London, UK, 1988, pp. 134—155.

[2] ASTM Standard C 1260, “Standard Test Method for Potential Alkali Reactivity of Aggregates (Mortar-bar Method),” *Annual Book of ASTM Standards*, Vol. 04-02, ASTM International, West Conshohocken, PA, 2006.

[3] Lu, D., Fournier, B., Grattan-Bellew, P.E., Xu, Z. and Tang, M., “Development of a Universal Accelerated Test for Alkali-Silica and Alkali-Carbonate Reactivity of Concrete Aggregates,” *Materials and Structures* (accepted: online first).

[4] Sims, I. and Nixon, P., “RILEM Recommended Test Method AAR-0: Detection of Alkali-Reactivity Potential in Concrete – Outline guide to the Use of RILEM Methods in Assessments of Aggregates for Potential Alkali-Reactivity,” *Materials and Structures*, Vol. 36, 2003, pp. 472—479.

[5] Wigum, B.J., Pedersen, L.T., Grelk, B. and Lindgard, J., “State-of-the Art Report: Key Parameters Influencing the Alkali-Aggregate Reaction” *SINTEF Building and Infrastructure Report SBF52 A06018*, Norway, 2006.

[6] Shayan, A. and Morris, H., “A Comparison of RTA T363 and ASTM C1260 Accelerated Mortar Bar Test Methods for Detecting Reactive Aggregates,” *Cement and Concrete Research,* Vol. 31, 2001, pp. 655—663.

[7] Oberholster, R.E. and Davies, G., “An Accelerated Method for Testing the Potential Alkali-Reactivity of Siliceous Aggregates,” *Cement and Concrete Research,* Vol. 16, 1986, pp. 181—189.

[8] Berube, M.A. and Fournier, B., “Canadian Experience with Testing for Alkali-Aggregate Reactivity in Concrete,” *Cement and Concrete Composites*, Vol. 15, 1993, pp. 27—47.

[9] Higginson, E.C., “The Effect of Cement Fineness on Concrete,” *Fineness of Cement*, *ASTM STP 473*, American Society for Testing Materials, 1970, pp.77—81.

[10] Tennis, P., “Portland Cement Characteristics – 1998”, *Portland Cement Association Concrete Technology Today (PL992)*, Vol. 20, pp. 1—3.

[11] Berra, M., Mangialardi, T. and Paolini A.E., “Testing Natural Sands for the Alkali Reactivity with the ASTM C 1260 Mortar-bar Expansion Method,” *Journal of the Ceramic Society of Japan*, Vol. 106, 1998, pp. 237—241.

[12] ASTM Standard C 204, “Standard Test Method for Fineness of Hydraulic Cement by Air Permeability Apparatus,” *Annual Book of ASTM Standards*, Vol. 04-01, ASTM International, West Conshohocken, PA, 2006.

[13] ASTM Standard C 305, “Standard Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency,” *Annual Book of ASTM Standards*, Vol. 04-01, ASTM International, West Conshohocken, PA, 2006.

[14] Fournier, B. and Berube, M.A., “Application of the NBRI Accelerated Mortar-bar Test to Siliceous Carbonate Aggregates Produced in the St. Lawrence Lowlands (Quebec, Canada). Part 1: Influence of Various Parameters on the Test Results,” *Cement and Concrete Research,* Vol. 21, 1991, pp. 853—862.

[15] Hooton, R.D. and Rogers, C.A., "Development of the NBRI Rapid Mortar Bar Leading to its Use in North America" *9th International Conference on Alkali-Aggregate Reaction in Concrete*, London, UK, July 1992, The Concrete Society.

[16] Owsiak, Z., "Microstructure of Alkali-Silica Reaction Products in Conventional and Standard Accelerated Testing," *Ceramics – Silikaty*, Vol. 47, 2003, pp. 108—115.

[17] Nixon, P. and Sims, I., "Testing Aggregates for Alkali reactivity: Report of RILEM TC-106," *Materials and Structures*, Vol. 29, 1996, pp. 323—334.

[18] Shon, C., Sarkar, S.L. and Zollinger, D.G., "Testing the Effectiveness of Class C and Class F Fly Ash in Controlling Expansion due to Alkali-Silica Reaction Using Modified ASTM C 1260 Test Method," *Journal of Materials in Civil Engineering*, Vol. 16, 2004, pp. 20—27.

TABLE 1—*Comparison of accelerated mortar-bar tests* [2–6].

	ASTM C 1260 (United States)	**CSA A23.2-25A (Canada)**	**RILEM AAR-2 (Europe)**	**RTA T 363 (Australia)**	**CHINESE AMBT**
Water-cement ratio (by mass)	0.47	0.44 - 0.50	0.47	Flow table	0.33
Cement Alkali (Na_2O_{eq})	Not specified	1.0 ± 0.1 %	High alkali recommended*	Not specified	1.50 %**
Cement-aggregate ratio	1:2.25	1:2.25	1:2.25	1:2.25	10:1, 2:1, 1:1
Aggregate size (mm)	0.15-4.75	0.15-5.0	0.15-4.0	0.15-4.75	0.15-0.80
Bar size (mm)	25 by 25 by 285	25 by 25 by 285	25 by 25 by 285 or 40 by 40 by 160	25 by 25 by 285	40 by 40 by 160
Curing prior to zero length measurement (after 24 h in mold)	24 h in water @ 80°C	24 h in water @ 80°C	24 h in water @ 80°C	48 h moist curing + 4 h in water @ 80°C	4 h in 1M @ 80°C
Storage solution	1M @ 80°C	1M @ 80°C	1M @ 80°C	1M @ 80°C	1M @ 80°C
Immersion period (day)	14	14	14	21	7
Reactivity Criterion	>0.2 % in 14 d (0.1 - 0.2 % potentially reactive)	>0.15 % in 14 d	>0.2 % in 14 d (0.1 - 0.2 % potentially reactive)	>0.1 % in 10 d (<0.1 % in 10 d but >0.2 % in 21 d slowly reactive)	>0.10 % in 7 d

**Reference cement (Na2Oeq>1.15 & 1.38 %)*

***Low alkali cement with KOH added to mix water*

TABLE 2—*Chemical composition of the portland cements produced from high and low alkali clinkers.*

(%)	CaO	Al_2O_3	Fe_2O_3	SiO_2	MgO	SO_3	Na_2O	K_2O	Na_2O_{eq}	LoI[*]	Fineness (m^2/kg)
High Alkali Clinker	63.5	5.7	2.4	20.4	2.4	3.3	0.2	1.1	0.91	0.7	392
Low Alkali Clinker	64.6	5.0	4.0	20.9	1.2	2.9	0.1	0.4	0.37	0.4	403

[*] *Loss on ignition*

TABLE 3—Fineness of the produced portland cements.

	Grinding Time (minutes)	Blaine Fineness (m^2/kg)
H3	47	306
H4	82	392
H5	155	505
L3	60	294
L4	120	403
L5	*	518

[*] *Low alkali clinker could not be ground beyond 450 m^2/kg Blaine. After 4 hours of grinding the materialwas sieved over a 75-µm (No. 200) sieve in order to remove bigger particles and obtain desired fineness.*

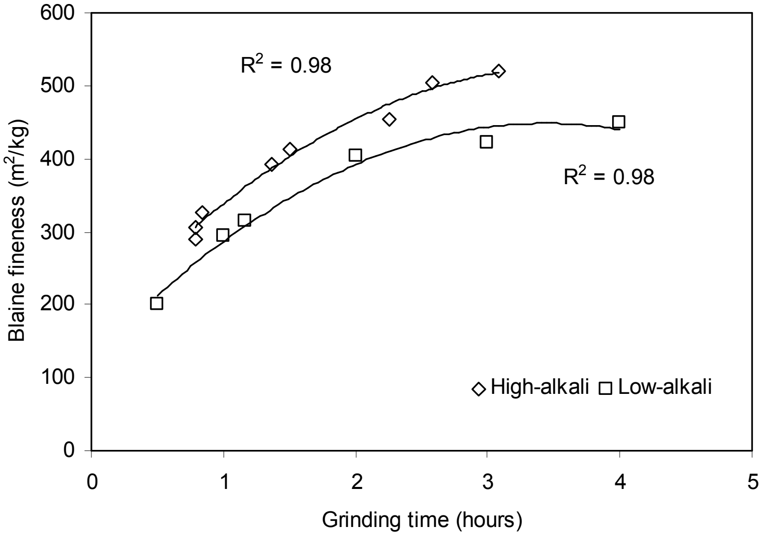


FIG. 1—*Grinding curves of the high and low alkali clinkers.*

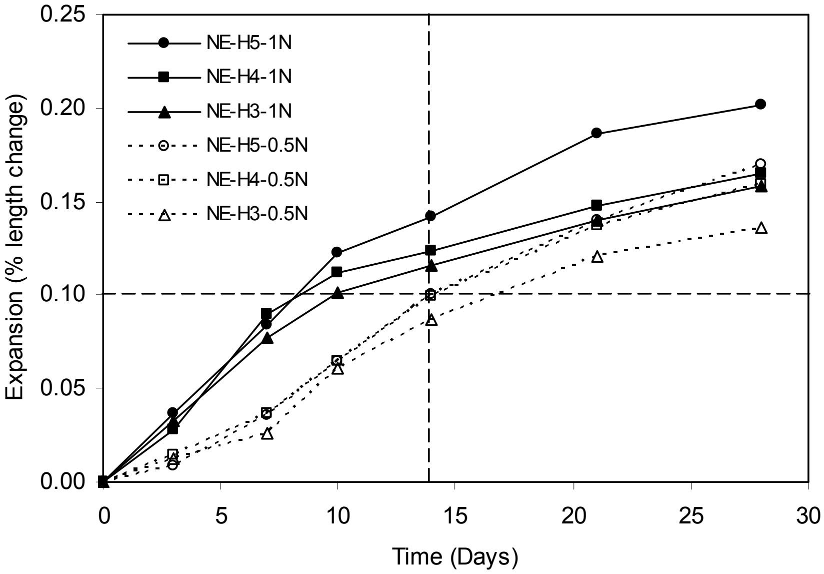


FIG. 2—*ASR expansion of moderately reactive aggregate with higher alkali clinker.*

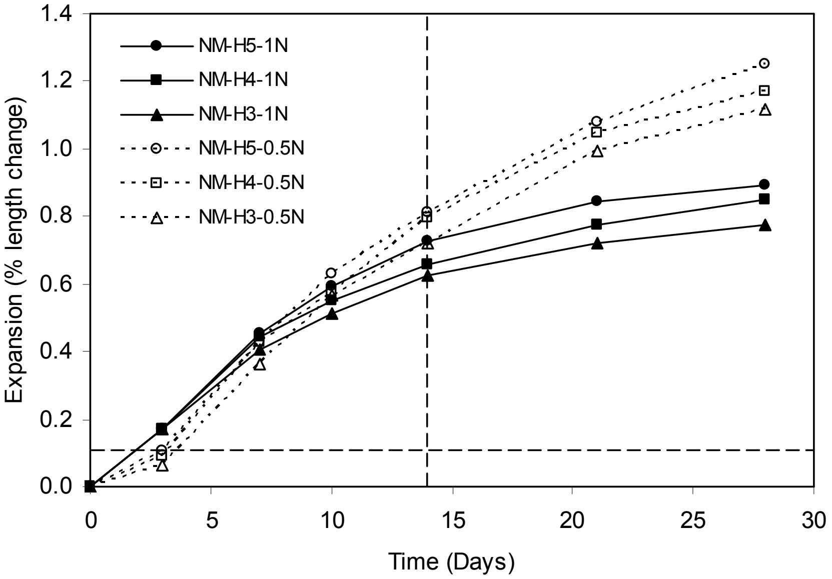


FIG. 3—*ASR expansion of highly reactive aggregate with higher alkali clinker.*

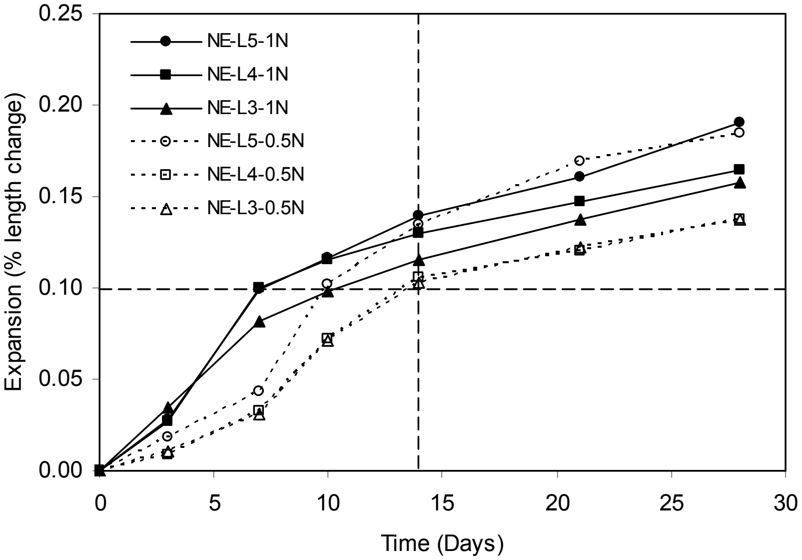


FIG. 4—*ASR expansion of moderately reactive aggregate with low alkali clinker.*

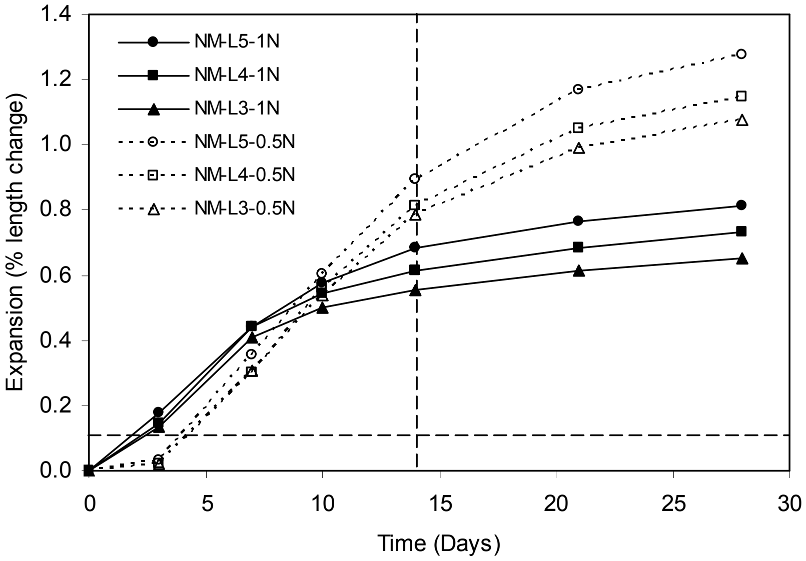


FIG. 5—*ASR expansion of highly reactive aggregate with low alkali clinker.*

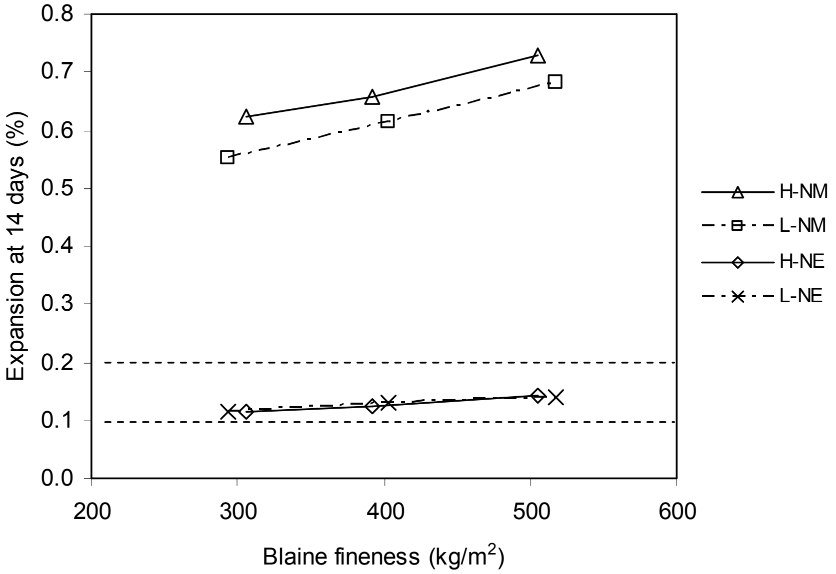


FIG. 6—*Fourteen-day expansions of the mortar-bars cast with high and low-alkali clinker cements.*

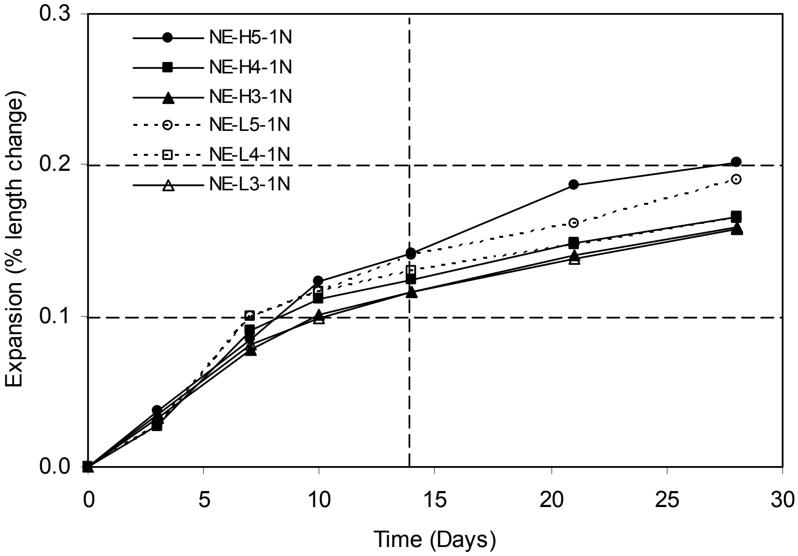


FIG. 7—*Expansion curves of the moderately reactive aggregate in 1N NaOH.*

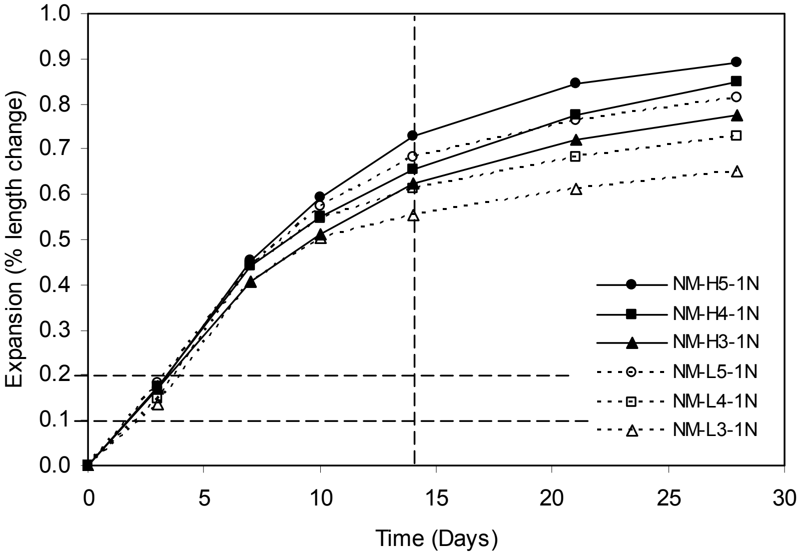
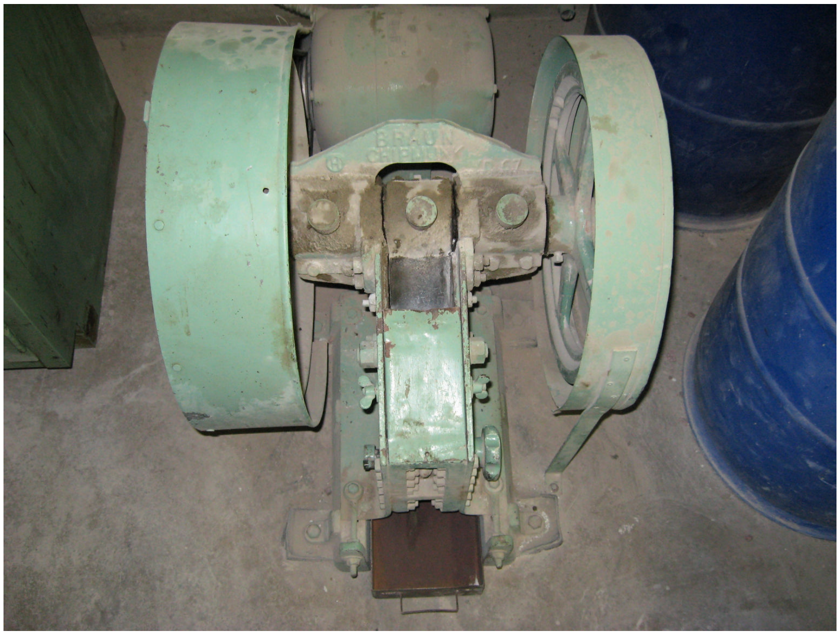


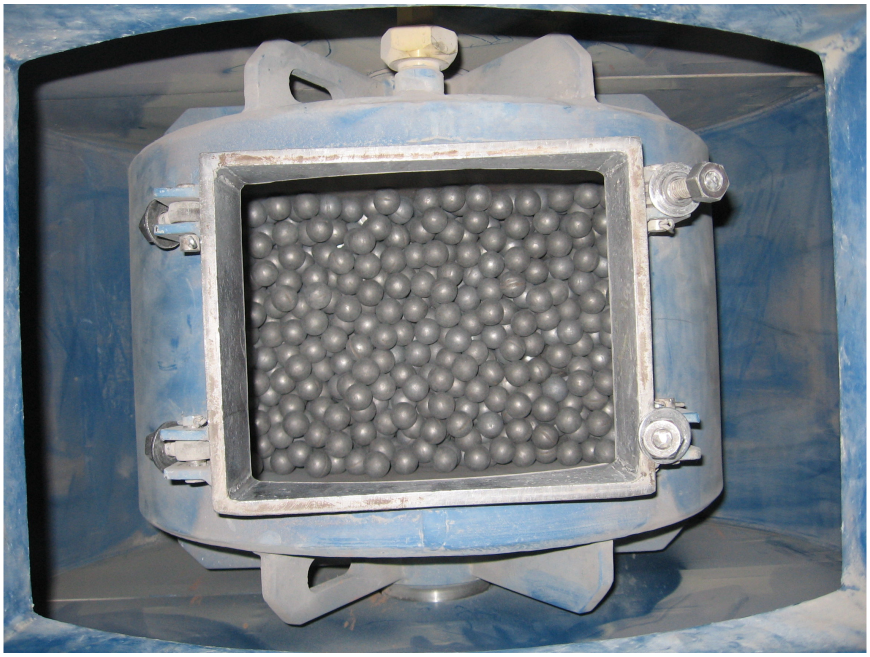
FIG. 8—*Expansion curves of the highly reactive aggregate in 1N NaOH.*

Appendix B: Pictures of Equipment



Jaw crusher used to reduce the size of crushed brick to pass 4.75 mm

Laboratory type ball mill with dust control casing



Steel balls inside the ball mill used to grind the crushed brick

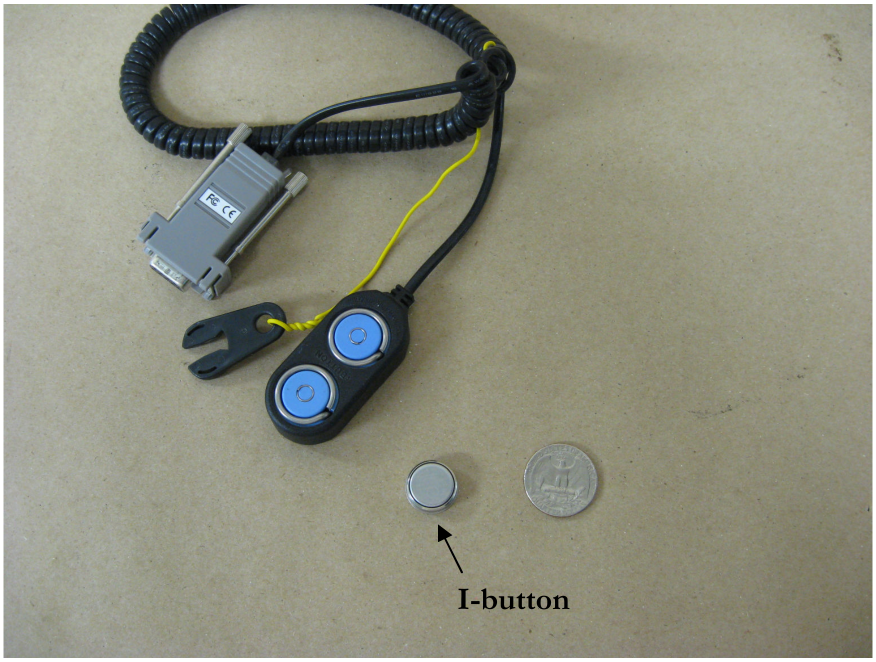
Various ball sizes for brick grinding – 40, 25, 12.5 mm

Blaine apparatus used to measure the fineness of portland cement

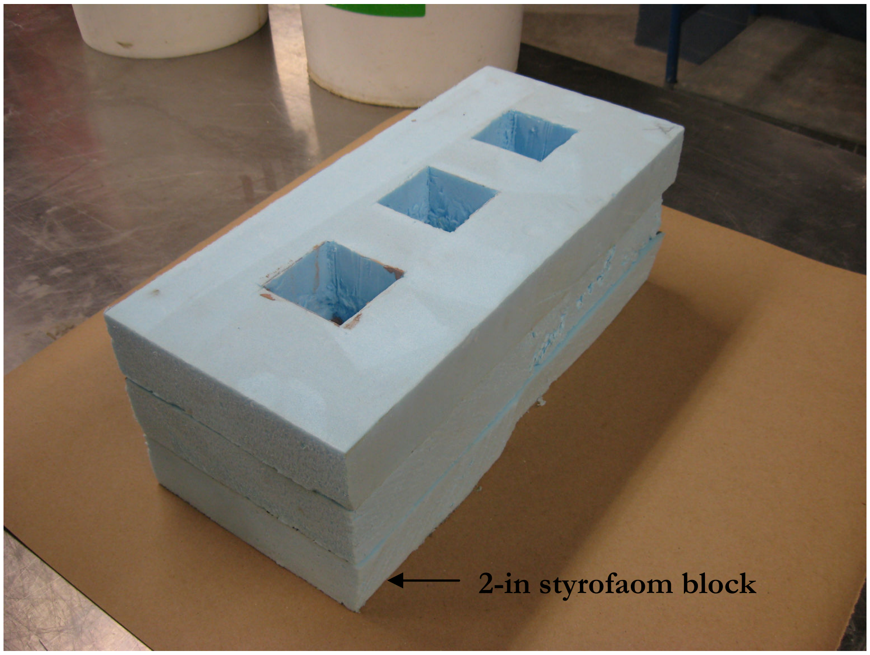
Water pressure regulator and ASTM No. 325 sieve for 45 µm residue analysis

Hobart mixer for paste and mortar preparation

Vicat apparatus for normal consistency and time of setting of cementitious pastes



I-button and data collector for temperature measurement during paste hydration

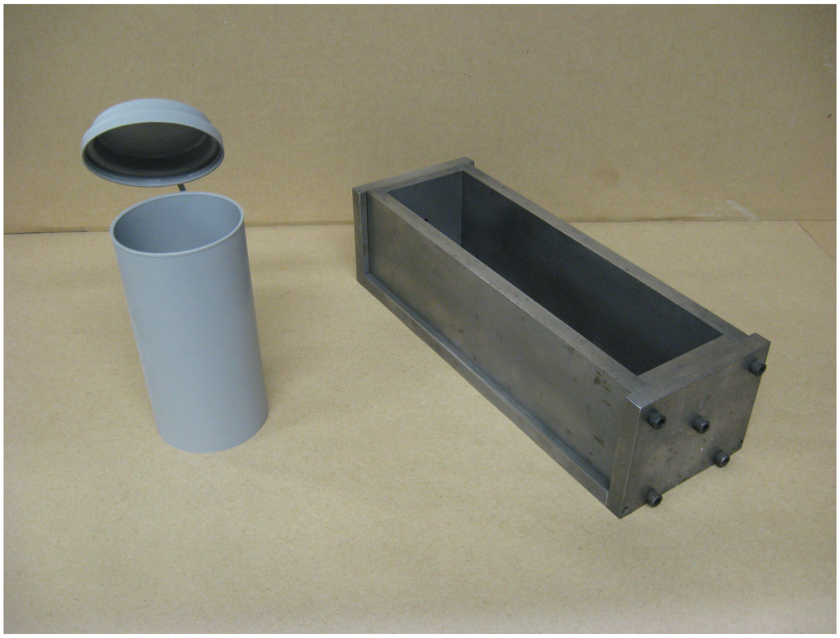


Insulated cells for paste samples in measuring temperature rise during hydration

Thermo-gravimetric analyzer (TA Instruments - TGA 2950)

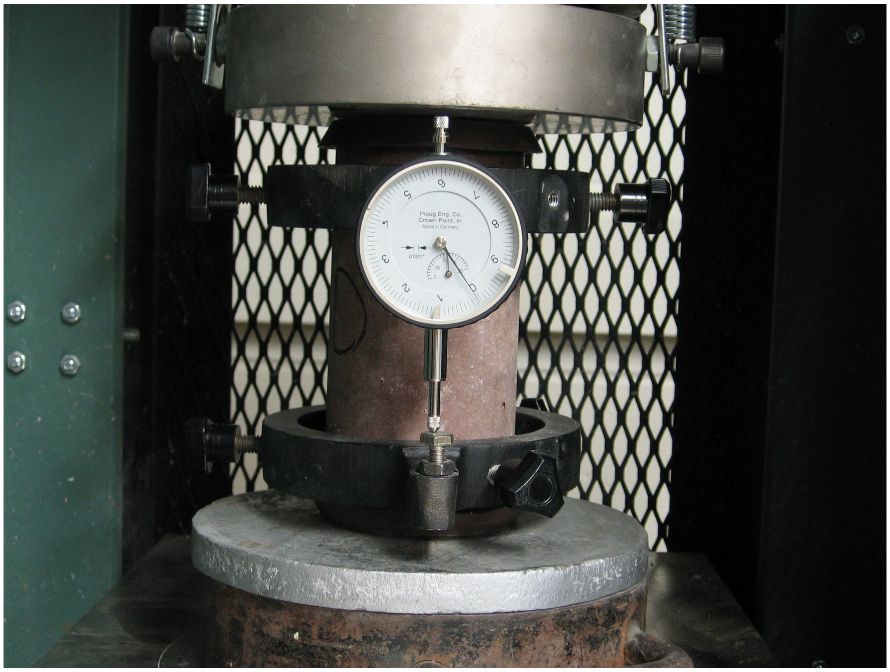
Concrete mixer – rotary drum type

Concrete mixer – pan type

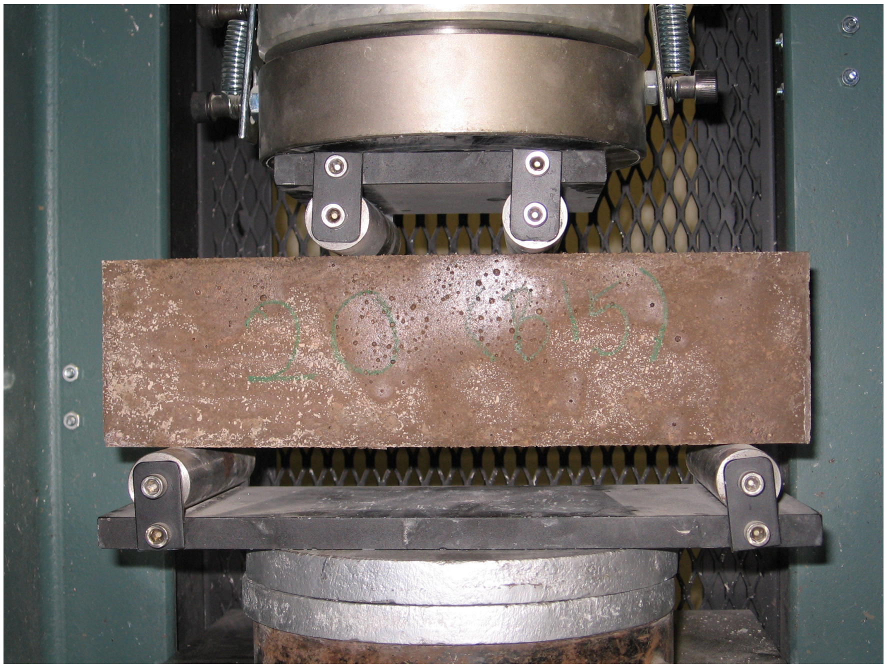


Disposable 75 × 150 mm (3 × 6 in.) plastic and 75 × 75 × 300 mm (3 × 3 × 12 in.) steel molds for testing compressive and flexural strength of concrete specimens, respectively

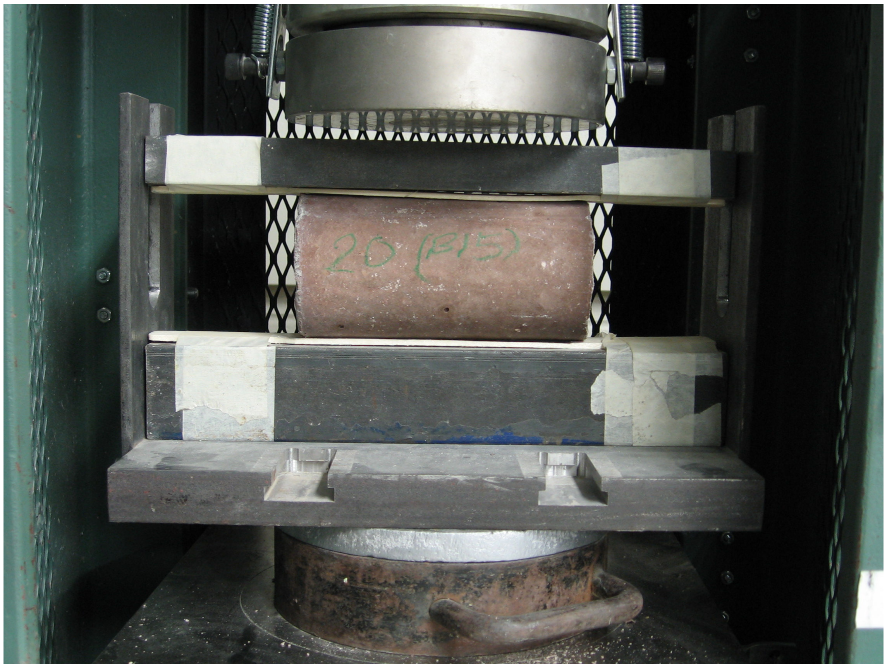
400,000 psi universal testing machine



Compressometer for modulus of elasticity determination of concrete



Apparatus for third point loading for flexural strength of concrete

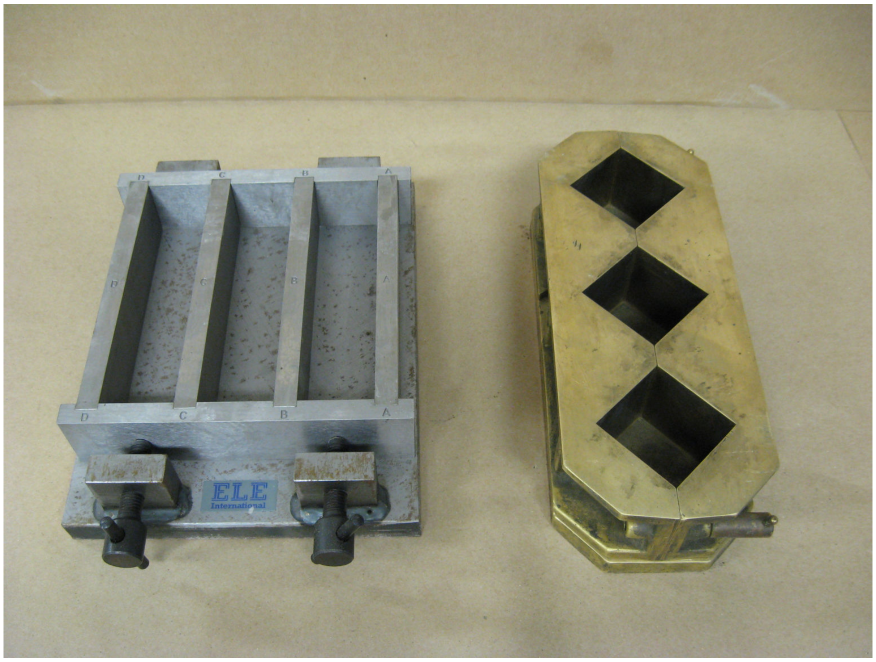


Configuration for splitting tensile strength determination of concrete

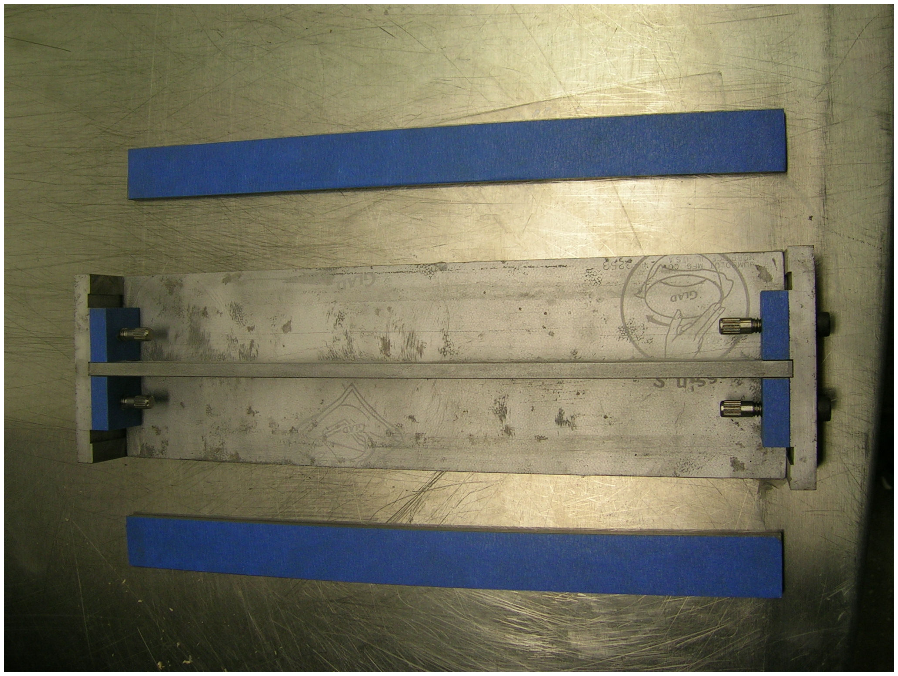
Concrete saw to cut 100 × 50 mm (4 × 2 in.) sections for rapid chloride permeability

Vacuum pump and desiccator for rapid chloride permeability testing

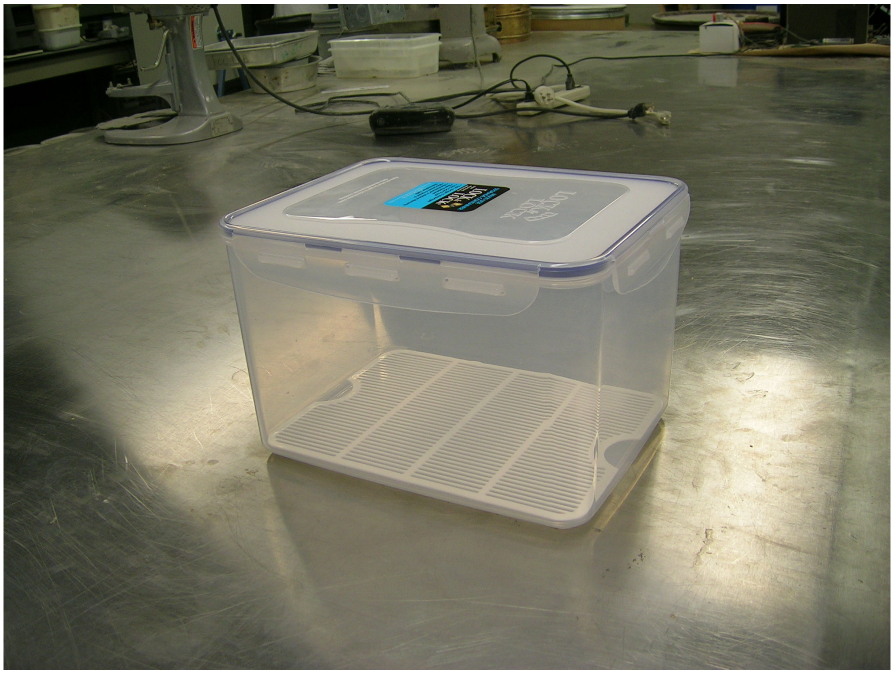
Permeability cells and data acquisition unit for rapid chloride permeability testing



40 × 40 × 160 mm (1.6 × 1.6 × 6.4 in.) steel and 50 mm (2 in.) brass cube molds for testing compressive and flexural strength of mortar specimens, respectively

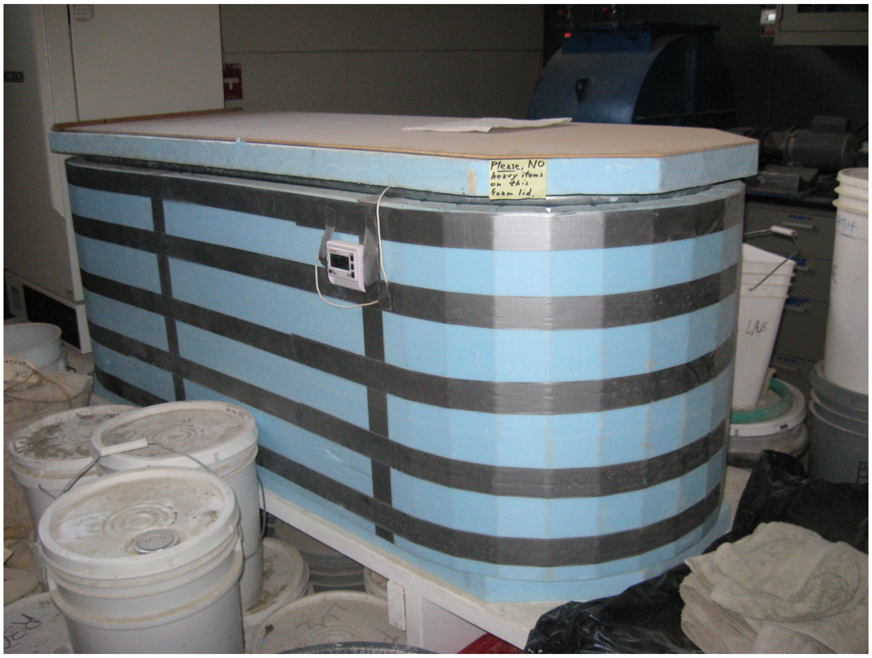


25 × 25 × 285 mm (1 × 1 × 11¼ in.) steel mold covered with releasable tape



Airtight container for the storage of mortar-bars in 1N NaOH solution at 80^{o}C

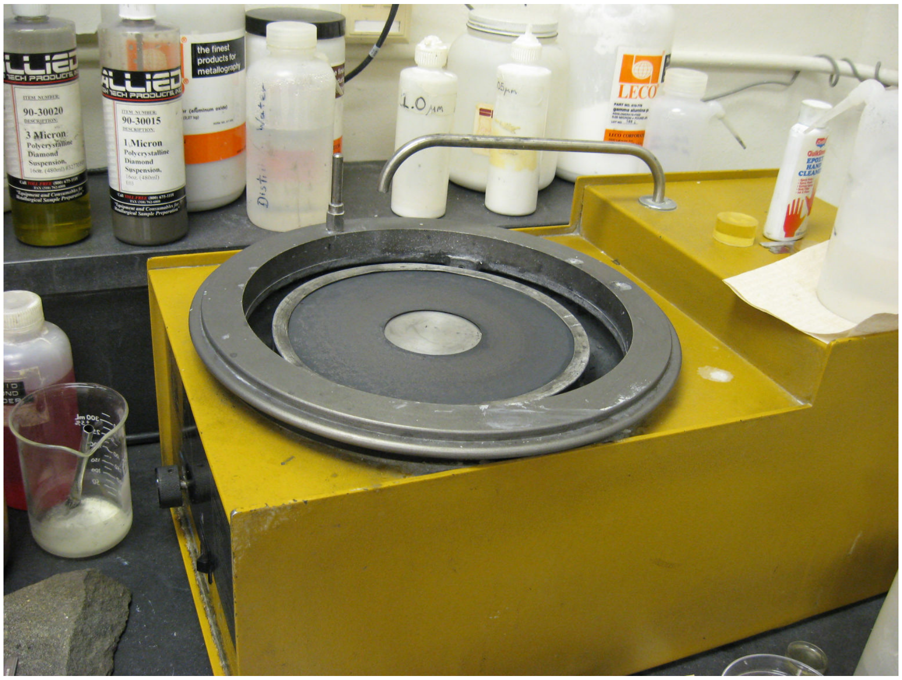
Comparator for length change measurement of mortar-bars and concrete prisms



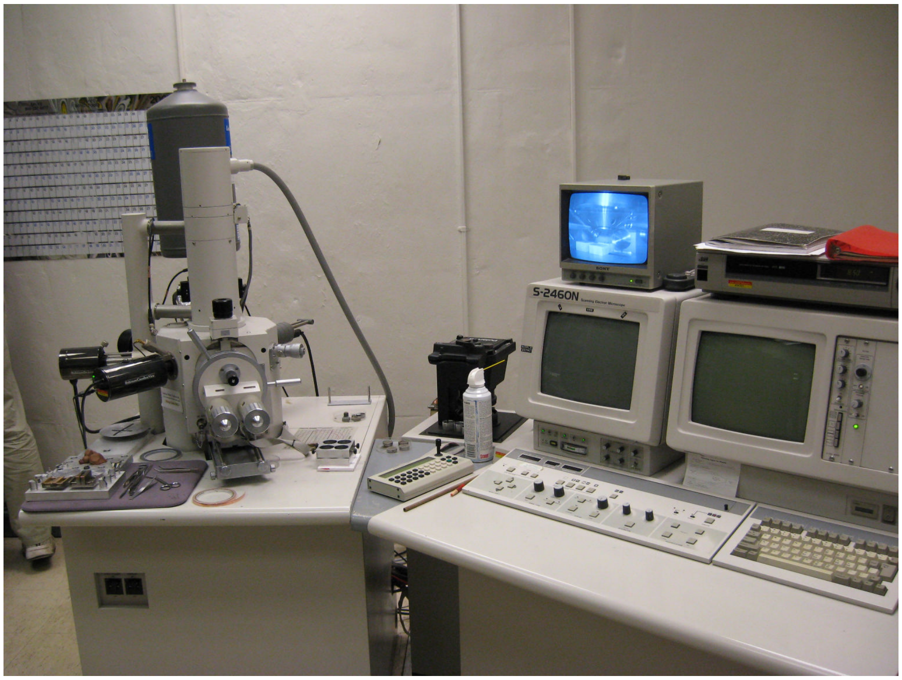
Curing tank for ASR conditioning (50^{o}C and >95% relative humidity)

Slow speed saw for detailed sectioning of concrete for microstructural investigation

Unit for epoxy impregnation under vacuum



Polishing wheel for SEM investigation



Scanning Electron Microscope (Hitachi S-2460N VP-SEM)

Appendix C: Vita

Vita

NAME OF AUTHOR: Fatih Bektas

DATE OF PLACE AND BIRTH: May 11, 1976, Karabuk, TURKEY

DEGREES AWARDED:

B.S. in Civil Engineering, Middle East Technical University, Turkey, 1994
M.S. in Civil Engineering, Middle East Technical University, Turkey, 2002
Ph.D. in Civil Engineering, Iowa State University, IA, 2007

HONORS AND AWARDS:

Premium for Academic Excellence (PACE) Award, *Iowa State University*, 2004
Portland Cement Association (PCA) Education Foundation Fellowship, *PCA*, 2005

PROFESSIONAL EXPERIENCE:

Research Asistant, Department of Civil Engineering, Middle East Technical University, Turkey, 1999-2004
Research Asistant, Department of Civil, Construction and Environmental Engineering, Iowa State University, IA, 2004-2007

PROFESSIONAL PUBLICATIONS:

F. Bektas, L. Turanli, K. Wang, H. Ceylan. *Comparative performance of ground clay brick in the mitigation of alkali-silica reaction*, J of Mater in Civil Eng (in press).
F. Bektas, T. Topal, M.C. Goncuoglu, L. Turanli. *Evaluation of alkali reactivity of cherts from Turkey*, Constr Build Mater (in press).
F. Bektas, L. Turanli, C.P. Ostertag (2006). *New approach in mitigating damage caused by alkali-silica reaction*, J Mater Sci 41, 5760-5763.
F. Bektas, L.Turanli, P.J.M. Monteiro (2005). *Use of perlite powder to suppress the alkali-silica reaction*, Cement Concrete Res 35, 2014-2017.
L. Turanli, B. Uzal, F. Bektas (2005). *Effect of large amounts of natural pozzolan addition on properties of blended cements*, Cement Concrete Res 35, 1106-11.
L. Turanli, B. Uzal, F. Bektas (2004), *Effects of material characteristics on properties of blended cements containing high volumes of natural pozzolans*, Cement Concrete Res 34, 2277-2282.
F. Bektas, L. Turanli, T. Topal, M.C. Goncuoglu (2004). *Alkali-reactivity of mortars containing chert and incorporating moderate-calcium fly ash*, Cement Concrete Res 34, 2209-2214.
L. Turanli, F. Bektas, P.J.M. Monteiro (2003). *Use of ground clay brick as a pozzolanic material to reduce the alkali-silica reaction*, Cement Concrete Res 33, 1539-1542.