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Development and Characterization of Gypsum-Based Binder

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ABSTRACT

Strategies were devised for development of inorganic binders with moderate levels of moisture resistance using primarily natural raw materials subjected to simple processing methods. The raw materials considered here included gypsum, lime, citric acid and soda ash. In this binder, gypsum and calcite (resulting from carbonation of lime) provide binding effects, and the presence of calcite provides improved moisture resistance when compared with gypsum used alone. Citric acid is used in this formulation as a set retarder that also favourably affects the crystalline structure of gypsum. The binder system was prepared by blending the raw materials in different proportions, which was then mixed with sand and water, placed at room temperature and 50% relative humidity, and subjected to early-age compression test in air-dried and saturated conditions. Promising formulations were further evaluated using the SEM, EDX and XRD techniques. The results verified partial achievement of the targeted compositions at an early age.

Keywords: Natural Materials, Simple Processing, Inorganic Binder, Moisture Resistance, Gypsum, Lime

INTRODUCTION

Modern buildings are generally constructed with materials such as Portland cement concrete, steel, timber, pre-fabricated gypsum panels, masonry units, plastics, composites, and insulating foams. Building systems incorporating these materials are designed to meet structural safety, fire resistance, energy-efficiency, quality of life, and durability requirements. Some construction materials used commonly developing nations include adobe, rammed earth, cob, stabilized soil, stone, brick, concrete block, lime, gypsum, natural pozzolans, biomass ash, vegetable fibres (and their fabrics/ropes), straw, bamboo, tree trunks/stems/leaves, corrugated sheets, Ferro cement and asphalt [1-15]. These indigenous construction materials are commonly used in the context of traditional building systems which provide basic shelter under regularly encountered load and environmental effects at minimum cost. Their inability to withstand extreme events (e.g., earthquakes and storms), however, has led to a significant disparity in death tolls caused by natural disasters in developing nations versus those in developed nations where modern building materials and systems are prevalent [16]. The research reported herein focused on development of an indigenous binder made with abundant natural raw materials and simple processing techniques for production of construction materials with performance characteristics that fill the gap between modern and traditional materials of construction.

Past efforts along the lines followed in this research have used alkali activated non-wood biomass ash as an inorganic binder for production of construction materials [17-18]. Other examples of developing indigenous inorganic binders have made use of abundant natural raw materials such as laterite clay and volcanic tuffs [19]. A common theme here is to avoid the high processing temperatures of Portland cement, that can be achieved only in industrial setting [20].

The work reported herein has been inspired by the historic effects aimed at development of moisture-resistant inorganic binders for construction applications. Some surviving ancient buildings point at the improvements made in the moisture resistance and durability of gypsum binders via introduction of calcium and aluminium compounds which transform into moisture-resistant calcite and ettringite. Parallel with gypsum, lime produced via calcination of limestone emerged as another binder used in construction of ancient buildings [21].

MATERIALS AND METHODS

The option selected for development of stable hydraulic binders based on gypsum emphasized enhancement of gypsum using lime (to transform into calcite over time) and citric acid or sodium carbonate (to control set time and further enhance material properties). Gypsum, sodium carbonate, citric acid and saponin were purchased in powder form from Sigma Aldrich with 99% purity. Hydrated lime was purchased from a local hardware store in Lansing (MI).

The gypsum-based binder formulations considered in the experimental program are introduced in Table -1. Natural sand satisfying the ASTM C33 requirements for fine aggregate was the sand used in these mixtures. The gypsum-based mortar mixtures were prepared in a mortar mixer (Hobart A-200). The fresh mix was cast into 50-mm cubes, stored at room temperature with 50% relative humidity, and demolded after 24 hours. After demolding, the specimens were stored at 50% relative humidity and room temperature; and were subjected to compression tests at 7 days of age. Two specimens were tested with their moisture content stabilized at 50% relative humidity, and two other were tested in wet condition after 24 hours of immersion in water at room temperature. Longer-term storage would have enhanced carbonation of lime which benefits the strength and moisture resistance of the specimens.

The microstructure products were evaluated via scanning electron microscopy using a JEOL JSM-6610LV scanning electron microscope (JEOL Ltd., Tokyo, Japan). EDX analyses were also conducted in the course of scanning electron microscopy. The mineralogy of the binder was assessed using the x-ray diffraction (XRD) technique. A Bruker D8 daVinci diffractometer equipped with Cu x-ray radiation operating at 40 kV and 40 mA was used for performance of the XRD tests at a rate of 5º/min, covering a reflection angle range 2θ of 10−80°.

Table -1 Mix Designs (Weight Ratios) Evaluated for Development of the Hydraulic Binder

Mix	$CaSO_4.½H_2O$	Lime	Additive	Water	Sand
1	1	0	0	0.6	3
2	0.8	0.2	0	0.6	3
3	0.5	0.5	0	0.6	3
4	0.8	0.3	0.025 (Na_2CO_3)	0.6	3
5	0.8	0.2	0.05 (Na_2CO_3)	0.6	3
6	0.8	0.2	0.02 (Citric Acid)	0.6	3

RESULTS AND DISCUSSION

The seven-day compressive strength test results (average of two specimens) for air-dried and wet specimens are presented in Fig. 1. It should be noted that early-age testing of specimens is particularly unfavourable to mixtures incorporating lime, because reaction of lime with carbon dioxide generates calcite over time, which enhances the mechanical performance and moisture resistance of these specimens. The 7-day test results presented in Fig. 1 indicate that:

- introduction of lime (at hemihydrate: lime ratio of 0.8: 0.2) yields beneficial effects (Mix 1 vs. Mix 2);
- increasing the lime content (from hemihydrate: lime ratio of 0.8: 0.2 to 0.5: 0.5) adversely influences the early-age strength of the gypsum-based binder (Mix 2 Vs. Mix 3);
- sodium carbonate is not an effective additive for enhancing the early-age strength of gypsum-based binders (Mix 2 Vs. Mixes 4 & 5);
- citric acid is an effective additive for enhancing the early-age strength of gypsum-based mortars, with a 7-day compressive strength of about 11.5 MPa achieved in air-dried condition with gypsum: lime: citric acid weight ratios of 0.8: 0.2: 0.02; and
- the viable gypsum-based mixtures developed in this investigation (Mixes 2 & 6) experienced about 50% loss of compressive strength upon saturation, noting that further generation of calcite via reaction of Portlandite with the carbon dioxide in air is anticipated to enhance the moisture resistance of these mixtures over time. The gypsum-based mix selected based on these test results comprises gypsum: lime: citric acid at weight ratios of 0.8: 0.2: 0.02. The 7-day compressive strength of 11.5 MPa exceeds those specified by ASTM C1157 for the low heat of hydration (LH), moderate heat of hydration (MH) and high sulphate resistance (HS) hydraulic cements.

Fig. 2 shows an SEM image depicting the surface topography of the refined gypsum-based binder (Mix 6). The needle-like structure of gypsum as well as the calcium hydroxide crystals and the sand particles can be identified in this image (Fig. 2a). The SEM image indicates that gypsum is the binding material in the young gypsum-lime mortar. Fig. 2b points at the integration between gypsum needles portlandite crystals. Longer curing duration and reacting with the carbon dioxide in the air could transform portlandite to more stable calcite.

The SEM image and EDX analysis depicted in Fig. 3 indicate that, besides calcium and sulphur, carbon is also a primary element which could point at carbonation reactions which would be even more prevalent at later ages. The resulting carbonates enhance the binding qualities and the moisture stability of the refined gypsum-based binder. Fig.

4 shows the XRD spectrum of the refined gypsum-based binder (Mix 6). Gypsum is observed to be the primary crystalline phase. Small amounts of calcite and portlandite were also detected in the binder. The purpurine hydrate peak is probably indicative of the use of citric acid as an additive (for set retardation and enhancement of moisture resistance) in this refined gypsum-based binder.

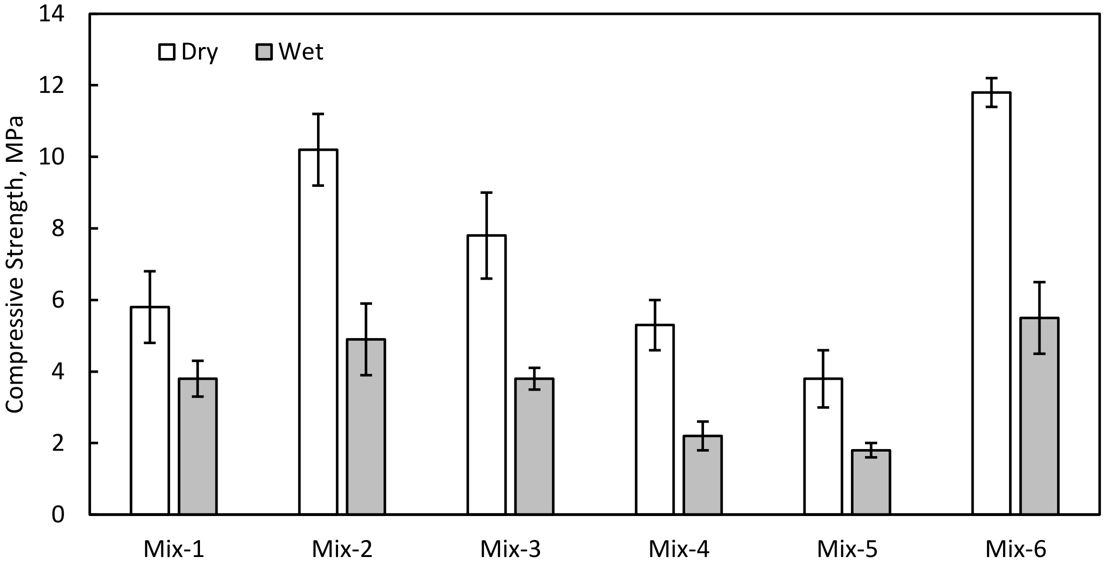


Fig. 1 Compressive strength test results for gypsum-based mortars at age of 7 days

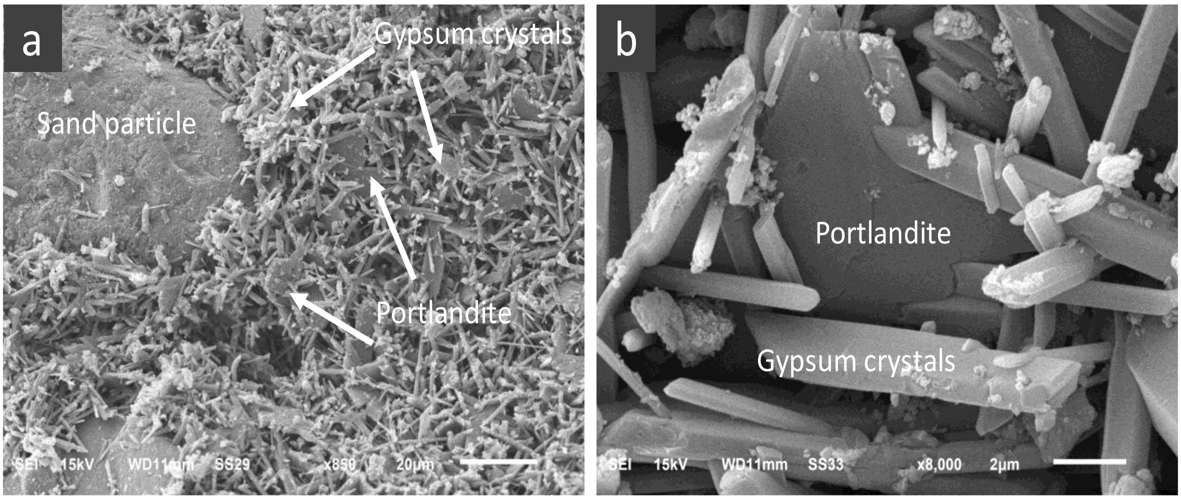
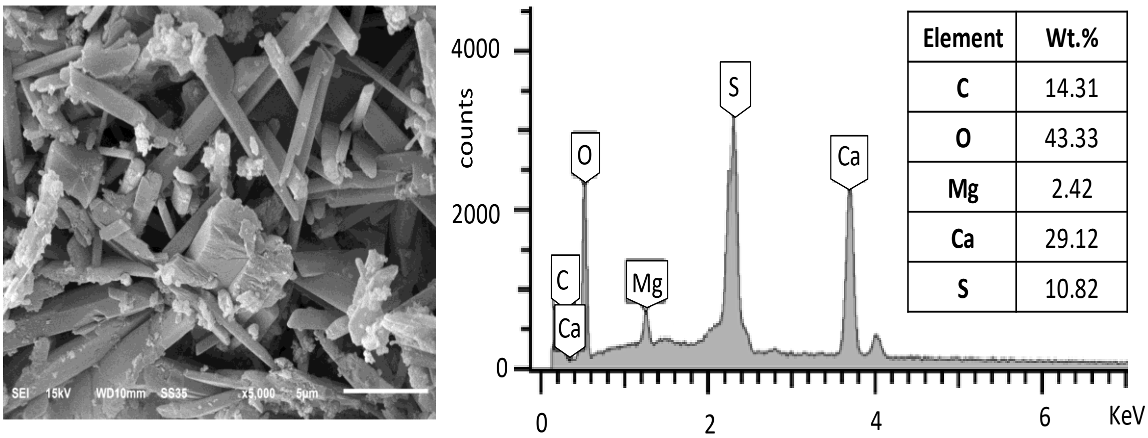


Fig.2 SEM image of the mortar with refined gypsum-based binder



Element	Wt.%
C	14.31
O	43.33
Mg	2.42
Ca	29.12
S	10.82

Fig. 3 Secondary electron images and EDS spectrum of the refined gypsum-based binder

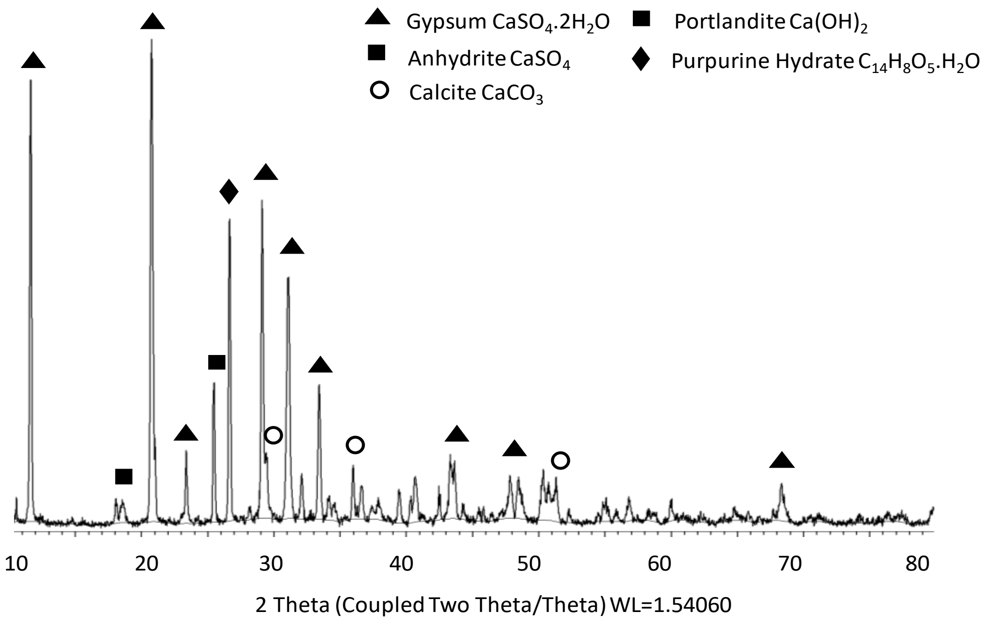


Fig. 4 X-ray diffraction spectrum of the refined gypsum-based binder

CONCLUSIONS

Gypsum is an abundant natural resource, which can be simply and sustainably converted into an inorganic binder. Gypsum binders, however, are moisture-sensitive. The approach evaluated in this work for enhancing the moisture resistance of gypsum involved its blending with lime (another indigenous binder) to produce Portlandite which transforms into a stable calcite binder via reaction with the carbon dioxide in air, and use of additives such as citric acid which control the rate of setting and alter the crystallization kinetids and the morphology of gypsum crystals.

Early-age compressive strength test results indicated that the addition of lime at 20% by weight of gypsum, and the use of citric acid at 2% by weight of the solid binder yielded viable levels of early-age compressive strength complemented with some level of moisture resistance. Testing at early age, however, did not allow for extensive carbonation of lime to significantly enhance the moisture resistance of the resultant binder. Microstructural, chemical and mineralogical analyses indicated that gypsum crystalline needles constitute the primary binder at early age; early indications of the evolution of Portlandite into calcite via carbonation reactions could be detected.

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