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Potential of Using Industrial Wastes for Production of Geopolymer Binder as Green Construction Materials

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HIGHLIGHTS

- The focus of this investigation was on development of a new sustainable class of hydraulic binder that relies upon the robust nature of alkali aluminosilicate binder chemistry to make value-added and large-volume use of industrial wastes of rice husk ash, municipal waste solid ash (MSW) and coal fly ash as primary raw materials.
- In an effort to produce a hydraulic binder that requires only the addition of water (in lieu of alkaline solutions in traditional geopolymerization), the blend of raw materials was subjected to mechanochemical processing.
- The binder chemistry used here yields hydration products with desirable capabilities for stabilization of heavy metals.

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ABSTRACT

Rice husk ash, Municipal Solid Incineration Ash and coal fly ash was served as aluminosilicate precursor in synthesis of one-part alkali activated geopolymer binder. These by-products industrial wastes were subjected to physio-chemical processing of materials (mechanochemical synthesis) together with different naturally originated sources of alkaline earth metal cations into one-part alkali activated (geopolymer) binder binders. The binder of hydraulic binders was assessed based on their strength development attributes and morphology with respect to silica content, diffusion of Calcium ions and compositions of Alkali activator. The particle size distribution, the chemical composition, mineralogy, bond environment, of binder particles and effective immobilization of heavy metals of hydrated binder pastes were also inspected. The results highlighted that the ternary blend of combustion ashes to produce hydraulic binders with strength development qualities and safe disposal as material for building construction.

1. Introduction

Large-volume use of industrial wastes for production of construction materials can divert large quantities of industrial byproducts from landfills for value-added use towards improving the sustainability and economics of infrastructure systems [1–3]. This practice can also mitigate the release of toxic leachates to the environment by stabilizing the hazardous constituents of some industrial byproducts.

The slate of fuels burned by power plants to generate electricity has been expanded beyond coal, to include biomass and municipal solid waste (among others). Combustion of these fuels generates solid residues (ash). Examples of these solid residues are coal fly ash, bottom ash resulting from combustion of municipal solid wastes, and biomass (e.g., rice husk) ash [4–6]. These ashes largely comprise the mineral constituents of each fuel. The combustion process also concentrates any heavy metal constituents of the fuel in ash, which should be addressed in development of applications for the ash.

The work reported herein develops an alternative binder chemistry using a blend of coal, biomass and municipal solid waste combustion ashes. The binder chemistry used here yields hydration products with desirable capabilities for stabilization of heavy metals. Brief introductions to coal fly ash, rice husk ash, and municipal solid waste combustion ash are presented below followed by a concise introduction to the hydraulic binder chemistry considered here. A sustainable mechanochemical approach was adopted for

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production of hydraulic binder, which is also briefly introduced in the following.

Coal fly ash is the ash separated from the flue gas of power plants burning pulverized coal [7,8]. It comprises fine spherical particles that precipitate from the stack gases. Coal fly ash is largely amorphous with desired reactivity; its chemical composition comprises largely of silicon, aluminum and calcium.

Rice husks (RHs) is a byproduct of paddy rice processing. It is a renewable fuel for generation of energy and weighs approximately 20% of the harvested paddy. Rice husk ash (RHA) is the solid residue resulting from combustion of rice husk for power generation [9,10]. Its chemistry is dominated by silica (90–95 wt%), which is largely amorphous [11] with residual carbon as the main impurity and other traces metal cations such as K and Ca. Rice husk ash assumes a porous microstructure which raises its reactivity by producing large specific surface areas. Silica can be crystalline or amorphous depending on the temperature of calcination and exposure time [12,13]. Amorphous rice husk ash is a high-performance pozzolan was used as a supplementary binderitious material (SCM) that can partially replace ordinary Portland binder in the concrete mix proportions [9,14]. Geopolymers have also been prepared with RHA as a source of amorphous silica; investigations in this area have addressed the effects of such factors as alkalinity, curing time and particle size distribution [15–17].

Energy-from-Waste (EFW) facilities recover energy from municipal solid waste in specially designed boilers to ensure complete combustion for efficient recovery of energy [18,19]. The EFW process produces an ash byproduct that comprises the bottom ash that remains after the combustion process and the residue that remains from emission control systems. A main aim for using beneficiary of incineration residues is in production of construction materials. Bottom ashes have been investigated as road subbase material, and as an alternative to sand and gravel [20]. Replacing primary resources by secondary raw materials compacts material cycles and lower natural resource depletion, therefore contributing to the recognition of a circular economy. Moreover, the embodied energy relevant to first manufacturing of recycled materials is usually less than that of primary materials. In addition to the consumption of sourced materials, the consequences of resource mining must be taken into consideration [21]. Utilizing of residual ashes and municipal solid waste (MSW) ash in building materials can be viewed as a practicable alternative to landfilling, considering the past success in recycling of different waste sources (e.g., blast-furnace slag, coal fly ash, bauxite fines, phospho-gypsum) [22,70–71].

Geopolymerization process is based on the natural stoichiometry reaction that arises between amorphous silica- and alumina-rich solids in high alkaline activator solutions at nearly elevated temperatures. This reaction yields a geopolymer material with high-order in terms of stability, its microstructure consist of an amorphous polymeric network with interconnected Si–O–Al–O– Si bonds [23–28,68–70]. The advantage of RHA as a source of reactive silica for geopolymerizaiton relate to its highly amorphous nature and also its high specific surface area. Adequate sources of alumina should be used together with RHA in order to achieve viable Si/Al ratios [29–32].

Mechanochemistry concerns chemical and physio-chemical changes of materials at all states of aggregation via input of mechanical energy. The intensity of mechanical energy required to render mechanochemical effects can be realized in various mills, including the ball mills that are currently used in binder production facilities [44]. Mechanochemistry is often viewed as a "green" approach to chemical processing because it avoids the use of solvents and elevated temperatures [72], and attempts to induce favorable physio-chemical phenomena using non-hydrostatic mechanical stresses that are generally applied at high rates [33–37]. At the atomic scale, the mechanical deformation of crystalline structural arrangements under stress can be considered as a distortion or modification of the coordination shells of individual atoms. In turn, atomistic processes can be generically described as local structural excitations [33–37]. These excitations pull the system away from thermodynamic equilibrium, which usually promotes a significant enhanbinder of chemical reactivity [38,39].

A technical feasibility of incorporation industrial waste materials employed in the current work as a supplementary binderitious material in concrete mix design has been reported in [61–63]. A ternary blend of Portland binder, industrial residual ashes and limestone powder were formulated to improve the properties of self-compacted concrete [64], and the use of a mixture of these ashes in addition to limestone and wood fibers led to improve the properties of lightweight concrete blocks [64–67].

The main sight of this investigation was on development of a new sustainable class of hydraulic binder that relies upon the robust nature of alkali aluminosilicate binder chemistry to make value-added and large-volume use of rice husk ash, MSW ash and coal fly ash as primary raw materials. In an effort to produce a hydraulic binder that requires only the addition of water (in lieu of alkaline solutions in traditional geopolymerization), the blend of raw materials was forced to undergo Mechanochemical processing. This hydraulic cement can be used upon further investigations for construction applications such as lightweight building and paving roads [73–76].

2. Material and methods

2.1. Materials

Class C coal fly ash was supplied as dry powder by the Lansing Board of Water & Light (LBWL) in Lansing, Michigan. RHA with more than 90% amorphous SiO_2 content was obtained by controlled calcination of rice husk at 600 °C for two hours, followed by ball milling for 30 min, resulting in an average particle size of 22.84 µm. Commercially available potassium hydroxide flakes (caustic potash), sodium carbonate were used as alkaline materials. (ASTM C778) graded standard silica sand was selected as fine aggregate to prepare mortar mixes.

2.2. Particle size distribution

The particle-size distribution (PSD) (generated through laser granulometry) of the milled municipal solid water (MSW) incineration ash used in this work is presented in Fig. 1. The median particle size was 5.4 µm, and its specific surface area was 60,942 (cm^2/cm^3)

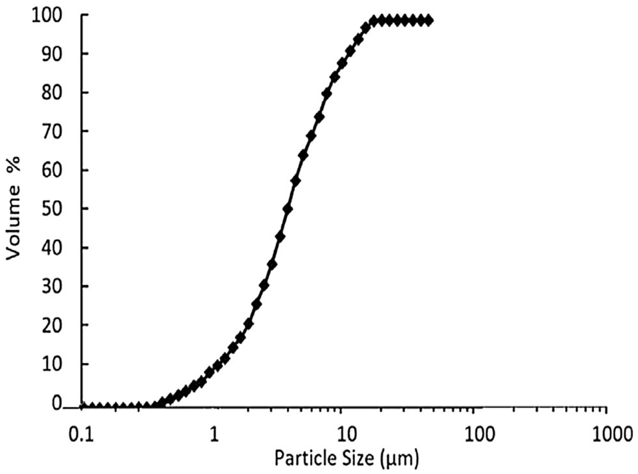


Fig. 1. Particle size distribution of the milled municipal solid waste (MSW) incineration ash.

Table 1
Chemical composition of coal fly ash, wt.%.

Material	SiO_2	CaO	Al_2O_3	K_2O	Na_2O	TiO_2	Cl	SO_3	LOI
Rice husk	90.0	0.60	0.01	2.30	–	–	–	–	5.10
Coal fly ash	43.05	14.30	23.33	1.72	0.90	0.03	–	–	1.82
MSW Ash	3.48	48.16	1.00	4.8	0.90	0.72	21.81	8.44	16.1%

2.3. Chemical composition

Table 1 introduced the chemical compositions of the ashes considered in this investigation, that were obtained via XRF spectroscopy. The MSW ash is observed to be rich in calcium, and also incorporate chloride and sulfur. Its loss-on-ignition is also relatively high. The mere presence of chloride and sulfur in MSW ash does not means that they would be necessarily available for deleterious reactions. They would not adversely influence the qualities offered by the resultant hydraulic binder if they get chemically bound in insoluble compounds. One should also evaluate the reason for the high LOI of MSW ash, considering that carbonates with no deleterious effects could also raise the LOI value. The high LOI values of MSW fly ash [41] and MSWI bottom ash [42] have been reported in the literature. The alkali content of MSW ash was more than those of other ashes considered in this investigation. The coal fly ash had relatively high silicon and aluminum oxide constitute that mainly comprised about 65% of the total mass, and the weight ratio of silicon to aluminum oxide was about 2. The relatively high (14.5%) CaO content of this coal fly ash qualifies it as Class C (high-calcium) per ASTM C618. Rice husk ash comprises largely of silica; its LOI value was moderate at 5.1%. The amorphous nature of rice husk ash makes it a valuable source of silica for synthesis of alkali aluminosilicate hydraulic binders.

2.4. Processing of raw materials, and characterization methods

The blend of coal ash, MSW ash and RHA was proportioned to produce a viable SiO_2/Al_2O_3 molar ratio of 2 to 4 which yields desired engineering properties. These formulations as well as those of alkaline materials considered in this investigation are summarized in Table 2. The blend of raw materials was undergoing mechanochemical processing to produce hydraulic binder as explained in previous investigations [40].

The effects of the relative proportions of rice husk ash and the municipal solid waste ash on the compressive strength development attributes of the resulting hydraulic binder were also investigated. The raw materials formulations considered in this series of tests are introduced in Table 3.

2.5. Characterization methods

The mortar mixtures had a silica sand-to-binder ratio of 2.5, and water-to-binder ratio of 0.5 to reach a desired fresh mix workability. They were prepared in a planetary mixer, cast into 50 mm cube molds, and consolidated under external vibration. The samples were kept in sealed condition at 90–97% relative humidity and at room temperature. They were demolded after 24 h and continued sealing at 90–97% relative humidity and room temperature (23C°) for compression testing at 7 days age. Compression tests were performed in universal test machine with speed of 0.1 mm/min. Ten reproduced samples were casted and tested for each mix design, and the average values of compressive strength were determined.

The chemical and elemental analysis of raw materials was inspected using X-ray fluorescence (XRF) spectroscopy. Their phase identifications were examined using X-ray diffraction (XRD) spectroscopy. FTIR spectroscopy was also performed on the raw materials and the hydraulic binder in the 4000–400 cm^{-1} range to observe their chemical properties (bond nature). The raw materials and the ruptured surfaces of hydrated binder pastes (at 28 days of age) were also evaluated via scanning electron microscopy to gain better understanding into their microstructural traits. SEM observations were made on a JCM-5000 NeoScope™ at an accelerating voltage of 10–15 kV using a secondary electron (SE) detector. Samples were sputtered with gold prior to SEM observations.

The Toxicity Characteristic Leaching Procedure (TCLP) is a test method used to determine whether a waste is hazardous due to its characteristics. TCLP tests were carried out to determine the potential for geopolymerization to reduce the leachability of specific elements in the IFA. The 20 heavy metals (Be, Al, V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Mo, Ag, Cd, Sb, Ba, Hg, Tl, Pb, and Th) were tested before and after mechanochemical processing.

3. Test results and discussion

3.1. Chemical bond environment

Fig. 2a presents the FTIR spectra of the raw materials for the coal fly ash, rice husk ash and MSW incineration ash. The FTIR data for the hydrated mechanochemically processed binder is presented in Fig. 2b. The distinct peak centered around 500–560 cm^{-1} (band A) can be assigned to the O–Si–O vibrational bending mode of the SiO_4 tetrahedra. This band provides an indication of the degree of “amorphization”, as the intensity is not associated with the degree of crystallization [43]. Peaks at infrared spectrum ranges of 1200–900 cm^{-1} (bands c, d) can be ascribed to the asymmetric stretching vibration mode of Si–O/Al–O peak at of 850–777 cm^{-1} (band f), that may be associated with asymmetric stretching vibration of Si–O–Si. The Si–O–Si stretching vibration mode is more influential than the O–Si–O bending mode. Therefore, one may use the Si–O–Si stretching vibration as an indication of the degree of geopolymerization [44–47]. The major differences between the as-received materials and the mechanochemically processed hydraulic binder relate to the shifts in the band around 1100–1200 cm^{-1} to around 1050 cm^{-1} (band g), and around 900 cm^{-1} (band c) to 870 cm^{-1} (band f), both of which decreased slightly in intensity. Moreover, the Si–O–Si peak shifted to a frequency that is lower than that in original residual ashes, indicating that a chemical change has been created in the binder phase. These shifts suggest that new binder/phases were created as a result of the

Table 2
The formulations (by weight) of raw materials considered for synthesis of hydraulic binders with different sources of alkalis.

Mix	RHA	Coal Fly Ash	MSW Ash	Alkaline Material	Water/Binder Ratio	Sand
1	0.3	0.2	0.1	Potassium hydroxide (0.4)	0.5	2.75
2	0.3	0.2	0.1	Sodium carbonate (0.4)	0.5	2.75

Table 3
The formulations of hydraulic binders considered for evaluation of the effects of the relative proportions of rice husk ash and the municipal solid waste ash on the compressive strength development attributes of the resulting binder.

Mix	RHA	Coal Fly Ash	MSW Ash	Alkaline Material	Water/Binder Ratio	Sand
1	0	0.2	0.4	Potassium hydroxide (0.4)	0.5	2.75
2	0.1	0.2	0.3	Potassium hydroxide (0.4)	0.5	2.75
3	0.2	0.2	0.2	Potassium hydroxide (0.4)	0.5	2.75
4	0.3	0.2	0.1	Potassium hydroxide (0.4)	0.5	2.75
5	0.4	0.2	0	Potassium hydroxide (0.4)	0.5	2.75

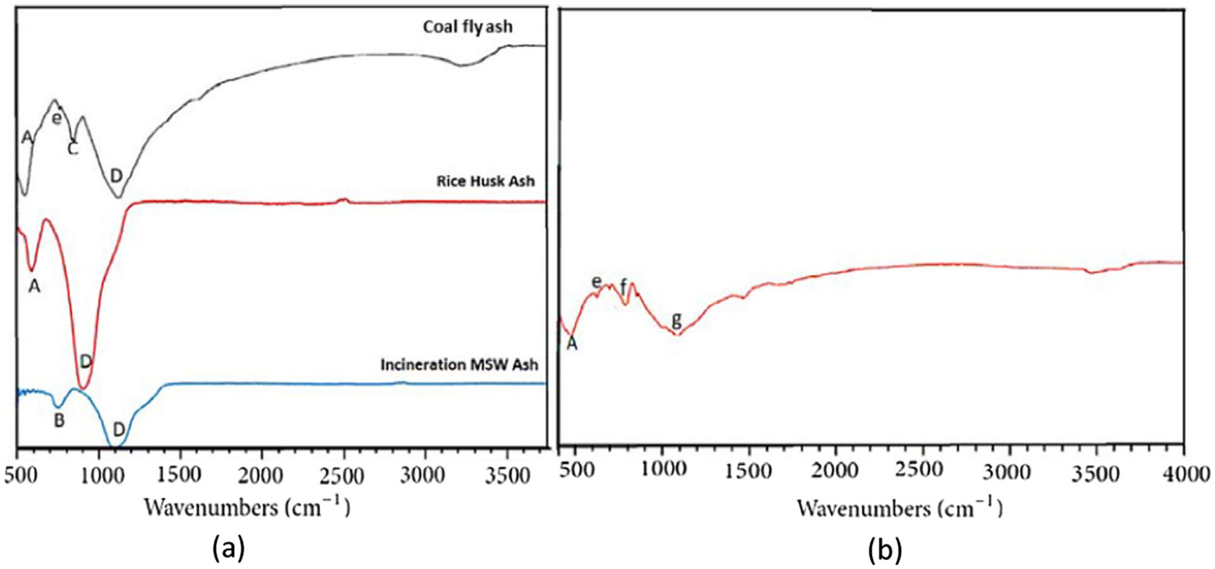


Fig. 2. FTIR spectra of: (a) raw ash materials; and (b) the mechanochemically processed hydrated binder.

mechanochemically induced reactions between ashes and alkaline materials (band c) [48]. This drop in intensity denoted that the amorphous phase in the residual ashes already have depolymerized to Si–O and Al–O bonds, and the shifts point at the polycondensation of these chemical bonds in the presence of alkaline medium [49].

3.2. Mineralogy

The scanning electron microscope images of different ashes presented in Fig. 3 indicate that: (i) the ground municipal solid waste incineration ash particles assume a random geometry with micrometer-scale dimensions, and appear to be dense; (ii) the coal fly ash particles exhibit characteristic spherical morphologies with micrometer-scale dimensions, and larger spherical particles seem to be coated with smaller particles; and (iii) ground rice husk ash exhibits a porous structure that increases its directly accessible surface area, and benefits its reactivity.

The XRD patterns of these ashes shown in Fig. 4 suggest that: (i) the municipal solid waste incineration ash is semi-crystalline, exhibiting XRD peaks corresponding to quartz and calcite, and it has a relatively low content of glassy phase when compared with other ashes; (ii) coal fly ash is also semi-crystalline with XRD peaks corresponding to mullite and quartz; and (iii) rice husk ash appears to be largely amorphous with a broad hump within around the region of 20-30° 2Theta that could point at the presence of an amorphized cristobalite [50]. The shift of FTIR spectra was observed in mechanochemically hydrated binder and it refereed to the formation of the new phases of binder (both Muscovite and Calcite, main elemental composition Ca and. This confirmed by X-ray powder diffraction analysis of the hydrated.

3.3. Compressive strength

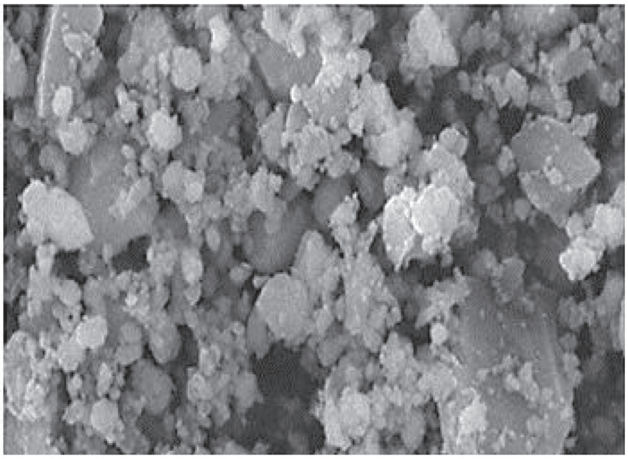
3.3.1. Effect of alkali source

The compressive strength test results obtained after 7 days of curing at room temperature in sealed condition are presented in Fig. 5 for geopolymer binders prepared with two different alkali sources (potassium hydroxide and sodium carbonate). Potassium hydroxide is observed to produce to produce a significantly higher 7-day compressive strength when compared with sodium carbonate. It should be noted that ASTM C1157 requires a 7-day compressive strength of 18.5 MPa for the 'General Use' class of hydraulic binder. The formulation and processing conditions considered in this experimental program yields a 7-day compressive strength exceeding 50 MPa, that is more than twice the standard requirement, when potassium hydroxide is used as the source of alkali. Sodium carbonate would be a successful activator as far as the formulation can readily release calcium cations to the solution in order to produce sodium hydroxide through reactions with sodium carbonate. In spite of the relatively high calcium content of the binder formulation considered here, its availability in solution could be why sodium carbonate could not act as a viable source of alkalis. The compressive strength obtained via geopolymerization reactions is influenced strongly by both the alumina-silicate precursors and the alkaline compound used in the formulation. The molecular structure and the concentration of alkalis are key factors influencing the geopolymerization process [51,52]. The OH ions supplied by alkalis (e.g., potassium hydroxide) attack the sialate-siloxo bonds to liberate aluminate and silicate to the solution that react to form rearranged gels, which finally condensate by releasing water to produce a hardened solid [53,54]. Strong alkalis tend to act more swiftly when the soluble silica is available, and play a crucial role in dissolving the silica and alumina constituents of the aluminosilicate precursors that enables their participation in the polycondensation stage of the geopolymerization process [53].

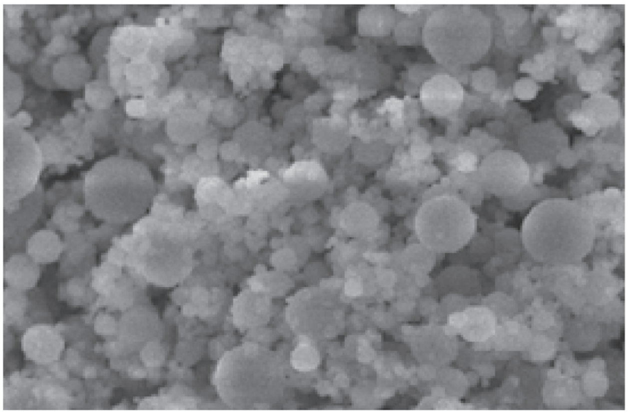
Scanning electron microscope images of the hydration products (Fig. 6) indicated that the non-reacted raw materials (e.g., spherical fly ash particles) are more notable in the case of binder prepared with sodium carbonate than that formulated with potassium hydroxide. This observation suggests that sodium carbonate could not, in the formulation considered here, could not produce the level of alkalinity required for effective dissolution of the aluminosilicate precursors.

3.3.2. Effects of the relative quantities of rice husk ash and municipal solid waste ash on the resulting hydraulic geopolymer binder performance

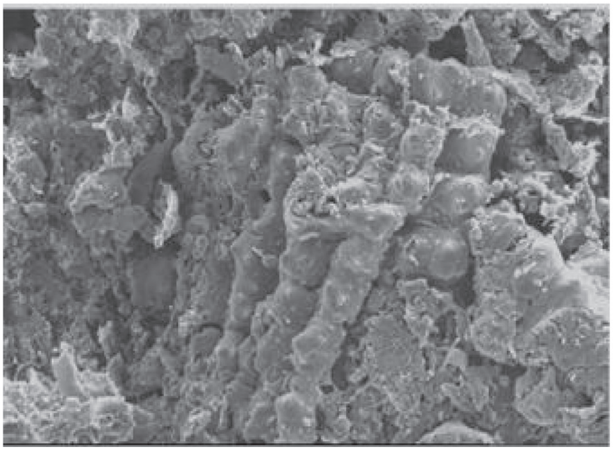
Rice husk ash is a source of reactive silica; the municipal solid waste ash, on the other hand, is rich in calcium oxide and incorporates some potassium oxide. They can feasibly make synergistic contributions to the chemistry of the hydraulic binder. An experimental investigation was undertaken in order to evaluate the



(i) Municipal solid waste (MSW) incineration ash



(ii) Coal fly ash



(iii) Rice husk ash

Fig. 3. SEM micrographs of the ground municipal solid waste (MSW) ash, coal fly ash, and ground rice husk used in this investigation.

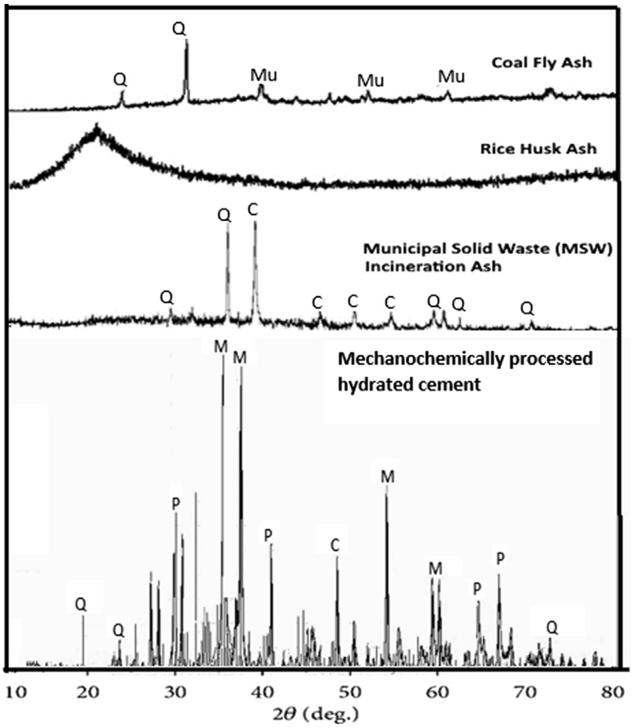


Fig. 4. XRD patterns of the three ashes used in this investigation (C = calcite, Q = quartz, Mu = mullite, and patterns of the mechanochemically processed hydrated binder; (Q: Quartz, M:Muscovite, P: Portlandite, C: Calcite).

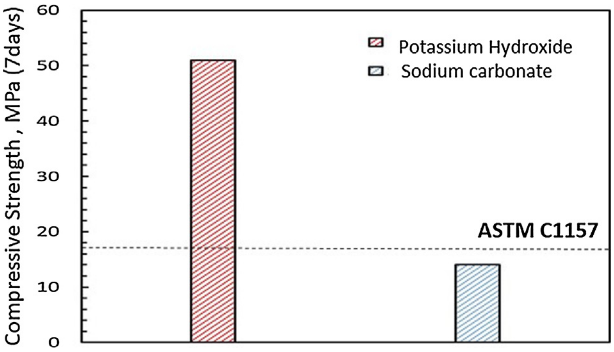
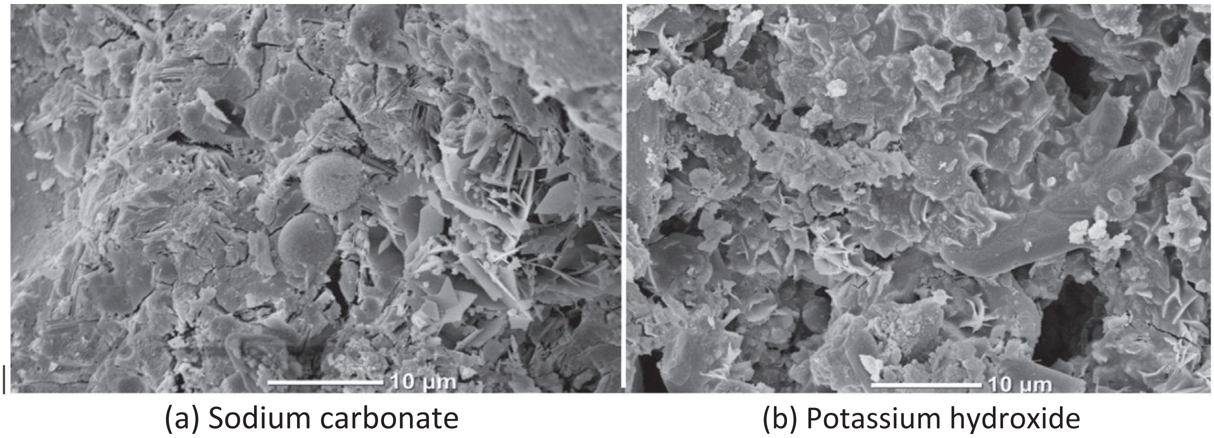


Fig. 5. Seven-day compressive strengths obtained with different sources of alkalis.

effects of their proportions in the hydraulic binder formulation on the end product performance.

Fig. 7 shows the compressive strength test data generated for the hydraulic binder prepared with different RHA and MSW ash contents. The compressive strength of all binder formulations is observed to increase with increasing RHA content (that is accompanied with decreasing MSW ash content). Rice husk ash (RHA) is a source of active silica; a rise in RHA content would thus raise the prevalence of Si-O-Si bonds which are stronger than either Si-O-Al and Al-O-Al bonds [23,28]. The reactivity of silica in RHA could also benefit the extent of reactions after similar curing conditions. A rise in RHA content is thus expected to raise the degree of condensation and the prevalence of relatively strong Si-O-Si bonds in the condensed tetrahedral aluminosilicate network, thus increasing the compressive strengths obtained with the hydraulic binder. Compressive strength, however, decreased when the rice husk ash content was raised beyond 30% (with 10% MSW content). This reversion of the trend in compressive strength with increasing RHA content could be attributed to: (i) hindrance of the reorganization of dissolved silica and alumina due to the excess concentration of soluble silica, which reduces the skeletal density of the geopolymer binder (reportedly occurs at Si/Al molar ratios exceeding 2) [55], which weakens the resulting gel; and (ii) reduced dissolution of rice husk ash particles, with the remaining unreacted or partially reacted rice husk ash particles (detected in the SEM



(a) Sodium carbonate (b) Potassium hydroxide

Fig. 6. Microstructures of hardened geopolymer binder pastes with either sodium carbonate or potassium hydroxide used as the sources of alkalis.

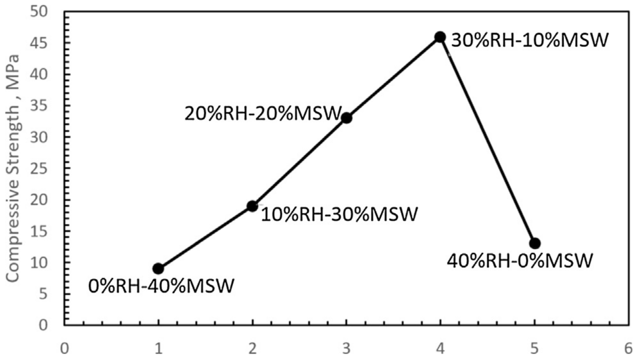


Fig. 7. Compressive strength development attributes of hydraulic binders formulated with different RHA and MSW ash contents.

images reviewed in the following) acting as porous fillers with adverse effects on strength [17]. For the raw materials types and formulations considered in this investigation, an RHA content of 30% (with an MSW ash content of 10%) seems to yield a satisfactory chemical stability in solution, inducing polycondensation effects with favorable strength development characteristics.

Fig. 8 presents representative SEM images taken from the fractured surfaces of the hydration products of binder's formulations with different RHA and MSW ash contents only – No coal fly ash content in this test-. Unreacted or partially reacted binder particles can be detected in all these images, which is a characteristic feature of some geopolymers [19]. A relatively dense structure with low amount of unreacted or partially reacted binder particles is noted for the binder formulation with 30% RHA and 10% MSW ash.

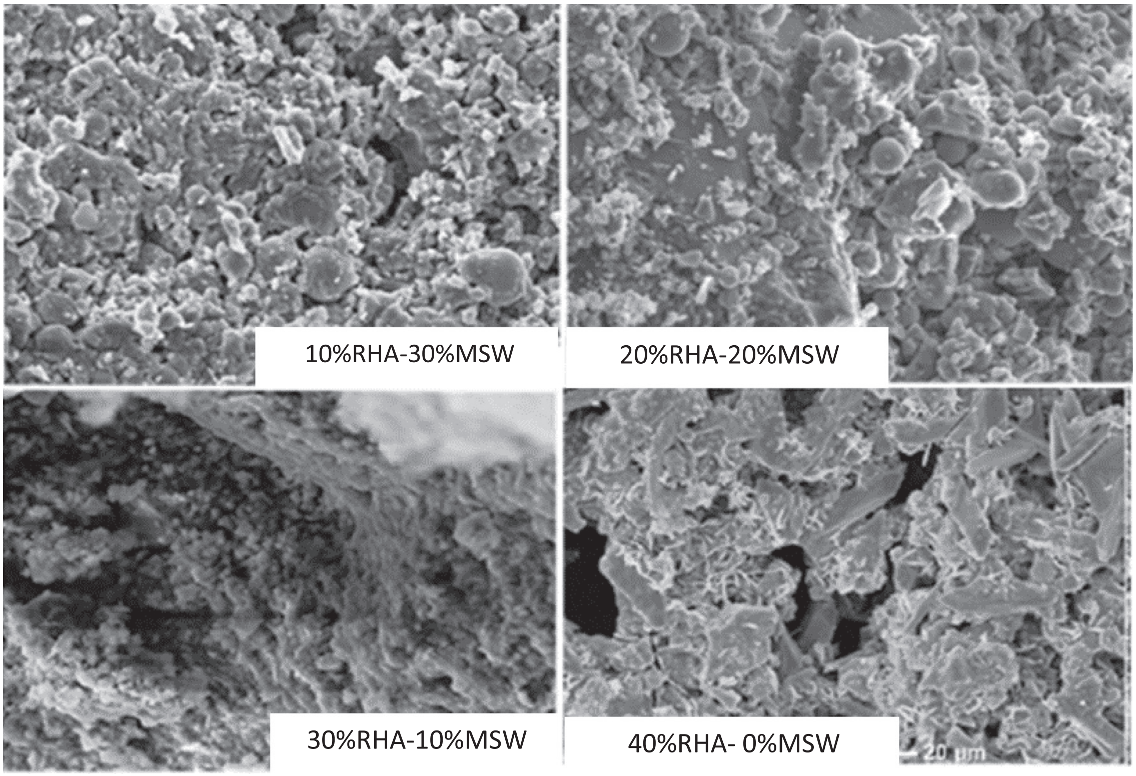


Fig. 8. Representative SEM micrographs of the hydration products of hydraulic binders formulated with different rice husk ash (RHA) and municipal solid waste (MSW) ash contents.

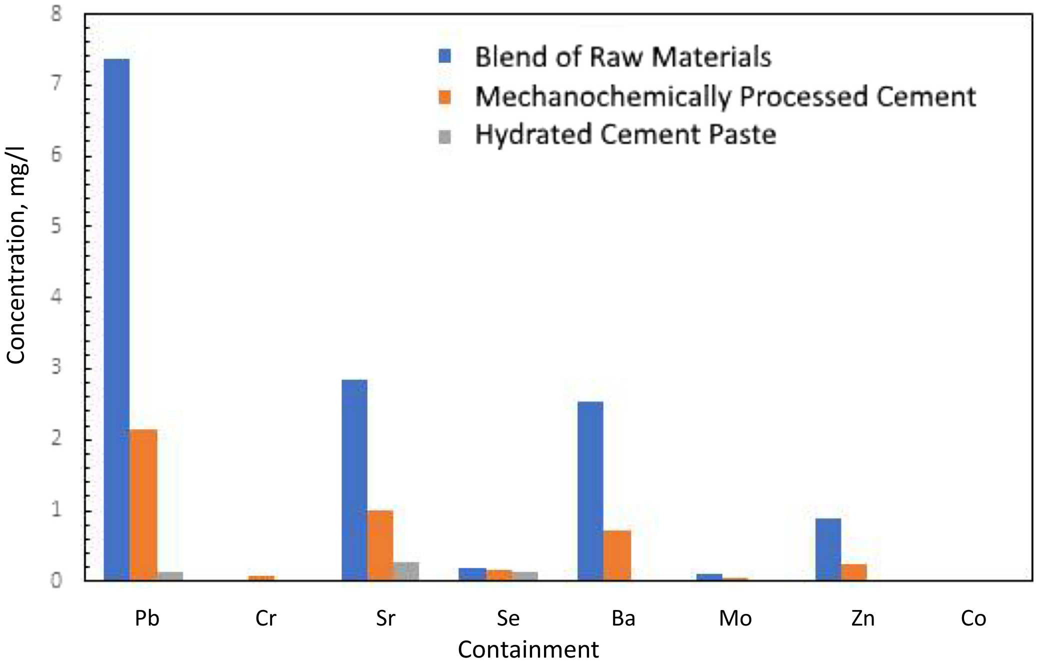


Fig. 9. Toxicity characteristic leaching procedure (TCLP) analysis of hydraulic binder prior to and after mechanochemical processing and subsequent hydration of binder.

3.4. Toxicity tests

TCLP tests were carried out to determine the potential of the mechanochemical processing and the subsequent hydration reactions to reduce leaching of heavy metals to the environment. Release of the RCRA heavy metals (Pb, Cd, Sr, Ba, Mo, Zn, Co) was evaluated for the blend of raw materials, the mechanochemically processed hydraulic binder, and the hydrated binder paste. The results for Formulation 4 (comprising 30% rice husk ash, 10% MSW ash, 20% coal fly ash and 40% KOH) are presented in Fig. 9. Mechanochemical processing produced significant reduction in the concentration of hazardous metal cations in leachates. This finding points at the immobilization capabilities of the physico-chemical phenomena that occur during mechanochemical processing of raw materials. Further drops in release of heavy metals are noted after hydration of the binder. This can be attributed to the release of Al and Si species in the alkaline solution, followed by geopolymerization reactions that yield structures with high heavy metals immobilization qualities [56,57]. The notable immobilization of Pb observed in Fig. 9 can be attributed to the formation of monomeric and/or oligomeric aluminate and silicate species, which then polycondense to form insoluble products that effectively encapsulate Pb. The immobilization of Pb within alkali activated matrices may be viewed as an encapsulation effect of the precipitates and gels, with limited contribution of the adsorption effects [58–60].

4. Conclusions

- Combustion ashes of rice husk, municipal solid waste and coal can be formulated to yield desired SiO_2/Al_2O_3 ratios and calcium contents for production of hydraulic binders with alkali aluminosilicate (geopolymer) chemistries. When blended with either potassium hydroxide or a blend of sodium carbonate and potassium hydroxide flakes, and subjected to mechanochemical synthesis, hydraulic binders with desired strength development characteristics can be obtained with this ternary blend of combustion ashes.
- There is an optimum silica content which maximizes the strength development attribute of the hydraulic binder. This silica content can be achieved by adjusting the rice husk ash content in the raw materials formulation. While higher contents of reactive silica (from rice husk ash) benefit the skeletal structure of alkali aluminosilicate hydrates, excess silica contents can disturb the polycondensation process and the resultant hydrates.
- The composition of alkali activator influenced the strength development characteristics of the mechanochemically processed hydraulic binder. The highest compressive strength was achieved when potassium hydroxide (in lieu of a blend of potassium hydroxide and sodium carbonate) was used as alkaline medium.
- Microstructural investigations of hydrated binder pastes confirmed that the density of hydrates and the extent of reactions of binder particles correlated with the compressive strength development characteristics of the hydraulic binders considered in this investigation.
- Mechanochemical processing of the blend of raw materials into a hydraulic binder reduces the presence of heavy metals among the leachates. Hydration of the resultant binder further reduces the leaching of heavy metals.

Declaration of Competing Interest

None.

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