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Potential Recycling of Metallurgical Slag Waste into Geopolymer Products: A Brief Review

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Abstract. Steel manufacturing process may produce waste such as slag, sludge, dust and mill scale. Since it is an unavoidable waste, the abundance of their production is becoming an environmental concern regarding the high rise of solid waste from the industry. There has been some well-known utilization of slag usage in the industry in handling the waste such as in road construction, cementing material as well as sorbent material. These past years had witnessed the ever-growing interest in the slag incorporation into geopolymer product due to their flexibility of the raw material range that makes incorporation of metallurgical slag waste possible. Thus, this paper seeks to review about the utilization of metallurgical slag waste into geopolymer product. The review focusing on the methodology, chemistry and the resulting properties of the geopolymers. Therefore, the paper will highlight on the characterizations of both raw materials and final product in terms of their microstructural, crystalline phase present and functional groups. Moreover, factors affecting the final product properties such as the alkali concentration, type of alkali, curing temperature, weight ratio and other factors that are mostly discussed by previous researchers are also critically reviewed.

1. INTRODUCTION

Steel manufacturing produces abundance of waste such as slag, sludge, dust and mill scale. Although slag is a by-product, it plays vital roles in steelmaking process. First, it gathers impurities during refining as preventive substance to the diffusion of N₂ and H₂ gases from atmosphere to the molten steel. It also protects molten steel against re-oxidation and barricade heat transfer from molten steel to the surrounding. Moreover, it is also a good emulsifier for hot metal to promote carbon oxidation [1]. Thus, the production of slag in the steelmaking process is unavoidable. However, too much waste production makes slag management becomes much more important to the manufacturer. Thus, large amount of steel waste increases the importance of slag stockpiling [2]. This means an increase in waste disposal costs which in return makes recycling economically interesting for the steel industry. The most being landfilled which increase the cost of landfilling and require large area of land [3]. Thus, the slag is being utilized in other application such as aggregate in road construction, cementing material, ceramic products as well as adsorbent for waste-water treatment.

Geopolymer has become very popular due to its superior advantages to other materials. First, the geopolymerization process occurs at much lower temperature (below 100°C), which results in low cost and energy saving during its production. Next, it reduces carbon footprint and thus serves as an alternative of ordinary Portland cement (OPC). Apart from possessing excellent early and long-term mechanical strength, geopolymers also have low shrinkage value, good fire and chemical resistance [4]. Geopolymer has found its used in various industries such as

treatment and containment of toxic waste, high-tech geopolymer resins and binders, fire- and temperature-resistant compounds, biotechnology, high-tech composites, low CO₂ geopolymer cements, concrete and ceramics [5].

In spite of the excellent properties, geopolymer provides the flexibility of being sustainable due to its source of aluminosilicate that can be used during the synthesis. Although the early production of geopolymer involve metakaolin as the main raw material, that fact is being changed by abundance of researches that are utilizing industrial waste to fully or partially replace metakaolin in the geopolymer synthesis. Those industrial waste such as fly ash, steel slag, mine tailings and many other are being incorporated as part of the aluminosilicate precursor of geopolymer. Thus, this paper seeks to review on the implications of incorporating the waste materials into the geopolymer synthesis.

2. SLAG IN GEOPOLYMERS

2.1 Blast Furnace Slag (BFS)

A considerable amount of literature has reported on utilization of Blast Furnace Slag (BFS) into geopolymer. In fact, BFS is the main type of metallurgical slag being used as aluminosilicate source in coating for polymer panel [6], geopolymer concrete repairing agent [7], adsorbent for Ni, As, Sb and sulphate removal from mine water [8,9], geopolymer grout [10] and geopolymer concrete [11].

Functional groups of the raw material and the geopolymer could be identified using Fourier Transform Infrared (FTIR) spectroscopy. BFS exhibited two small bands at 685cm⁻¹ and 1486cm⁻¹ which are respectively referring to the Al-O vibration and the asymmetric stretching of the O-C-O bonds. There are also bands at 900cm⁻¹ representing asymmetric stretching vibration of Si-O. The presence of double bands at 855 and 875 cm⁻¹ indicates the bending vibration of aragonite and calcite respectively. After geopolymerization, the FTIR spectra shows that the Al-O band (685cm⁻¹) was shifted towards lower value at 669cm⁻¹ indicating that the AlO₄ group was reacted which led to the formation of Al-O-Si bonds. Those at 855 and 900cm⁻¹ disappeared in the geopolymer spectra accompanied by appearance of a new intense peak at 973cm⁻¹ due to the formation of a new aluminosilicate structure based on the formed oligomers in the aqueous phase. The characteristic band of the calcite in BFS at 875 cm⁻¹ persisted after the geopolymerization process. This fact indicates that BFS particles were partially reacted unlike metakaolin which shows high reactivity. The small displacement of the O-C-O asymmetric stretching band at 1486 cm⁻¹ in BFS spectra towards 1493 cm⁻¹ corresponds to the formation of calcium carbonate or hydrotalcite [12]. There are strong absorption peaks between 3000cm⁻¹ and 4000cm⁻¹ due to the presence of -OH in BFS incorporated geopolymer. The bending vibration of the H-O-H bonds indicates that the geopolymer contains a fraction of free-state water, which was adsorbed on the surface or in the large cavity of the geopolymer. The asymmetrical stretching vibration peaks between 900 cm⁻¹ and 1000 cm⁻¹ indicate that the Si-O and Al-O bonds were the main products, which proves that the microstructure of the geopolymer is dominated by Si-O-Si (Al) bonds [13].

The crystallographic properties of BFS are investigated using XRD analysis. BFS has a mostly amorphous phase and a small amount of gehlenite. From the diffraction peak, a wide and weak peak appears at approximately 25-45° 2θ, which indicates that BFS is an amorphous material. The XRD patterns of the geopolymer shows a large diffusion halo diffraction peak at 20-40° indicating the composition of amorphous materials [6,13]. According to microstructural study by Jafari Nadoushan and Ramezaniapour (2016), it is found that the internal structure of the geopolymer is very compact, which ensures that the geopolymer has good mechanical properties [14]. Another study by Huang et al. (2016) have reported the presence of white colloid covering the surface of the geopolymer, which may be the product of the hydrated CSH gel [13].

Compressive strength of the geopolymer product was extensively highlighted by several researchers. They have suggested that the slag helps to increase the compressive strength when added in geopolymer production. Tennakoon et al. (2016) have discovered that the compressive strength of geopolymer increases with higher slag content regardless of the type of alkaline activator [11]. This is because, the double charged calcium ions introduced by slag combines with hydroxyl to form hydroxide precipitates followed by decrement in the pH reading around the hydroxide precipitates. Large solid particle of silicate polymers with lower surface energy are easier to form with the pH reduction. As a result, the nucleation and polymerisation between the soluble silicate and solid particles is improved and increase the rate of hardening. The blending of slag not only changes setting characteristics but also significantly alters the mechanical properties of the resulting geopolymer [11]. Huang et al. (2016) have suggested that BFS in alkaline conditions causes Si-O and Al-O of amorphous SiO₂ and Al₂O₃ to break and form oligomeric silicate ions and aluminium ions through the depolymerization reaction which generates an -O-Si-O-Al-O- skeleton of zeolite class polymers through a polycondensation reaction [13]. In the meantime, Huang et al. (2016) also have supported that the

CaO in the BFS and water generate a $\text{Ca}(\text{OH})_2$ solution. Silicate ions and aluminium ions combine with Ca^{2+} and H_2O and form a small amount of calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) gel. After that, the geopolymeric gel undergoes polycondensation to form a network gel with a larger molecular weight. Then, overall composition of CSH and CAH gel are created with hydrogen bonds which gives good mechanical properties to the geopolymer.

2.2 Electric Arc Furnace Slag (EAFS)

Usually, EAF slag can also be referred to as steel slag (SS). Bai et al. (2018) and Furlani et al. (2018) have investigated on the mix of EAFS and clay geopolymer [15,16]. Moreover, Nikolić et al. (2016) have studied on leaching behaviour of EAFS in alkaline solution of sodium hydroxide which affect the geopolymerization of EAFS [17]. In addition, Hoy et al. (2018) have discussed on utilization of EAFS geopolymer in recycled asphalt pavement (RAP) geopolymer [18].

FTIR spectra of pure steel slag has shown the presence of O-H stretching vibration peak related to structural and free water at 3445cm^{-1} and H-O-H bending vibration absorption peak at 1639cm^{-1} related to free water. This indicates that there were adsorption water and crystallization water in the steel slag powder, and the same phenomenon exists in the steel slag mixed with metakaolin geopolymer. The incorporation of steel slag causes the two peaks of regular metakaolin geopolymer to shift from 1021cm^{-1} (Si-O-Al) to 1015cm^{-1} and 706cm^{-1} (O-H) to 697cm^{-1} due to the relatively high iron and manganese contents in steel slag, and their ions have strong polarization ability and great affinity for oxygen. Characteristic absorption peak at 1420cm^{-1} in pure steel slag, whose strength decreased, and a certain displacement occurred in steel slag modified geopolymer (SSMGP) is attributed to the physical and chemical interaction between the steel slag and the geopolymer gel, which have weakened the lattice deformation and vibration of the steel slag [15]. The major bands lie in the $970\text{-}1160\text{cm}^{-1}$ range corresponding to the asymmetric stretching vibration of Si-O-Si and the Si-O-Al bonds. The intensive band at 1434cm^{-1} is related to the modes of CO_3 from CaCO_3 which is further confirmed by XRD analysis. It has been reported that this main carbonation band also has a peak at 874cm^{-1} which is very intensive and sharp peak. Due to the unreactive nature of this crystalline phase, it stays present after alkaline activation. The peak that indicates the presence of quartz is observed at 796cm^{-1} . Peaks at approximately 1418cm^{-1} and 990cm^{-1} correspond to the Si-O vibrations from potassium silicate, which reduce as the length of the process increases. The major band appear at $900\text{-}1100$ range, is assigned to the vibration of Si-O-T (T=Si or Al) bonds [19].

Bai et al. (2018) have conducted XRD analysis onto geopolymer incorporated with steel slag. They found that the active ingredient $2\text{CaO}\cdot\text{SiO}_2$ crystallization diffraction peak disappear. This suggesting that there is a certain degree of physical and chemical reactions between the steel slag components and the geopolymer matrix, as well as the lattice change or the decrease of crystallinity. Therefore, the bonding strength of the steel slag component and the geopolymer matrix is enhanced. A wide diffraction peak nearby 29° (2θ), indicating that the geopolymer is formed by the amorphous three-dimensional network structure [15]. From SEM micrographs of EAFS reported by Bai et al. (2018), they found that the shape of steel slag is irregular, and its pointedness is obvious with a small amount of agglomeration between small particles. When 10 wt.% of EAFS is mixed with metakaolin, the cracks are significantly reduced. This could be due to there were some active ions or groups on the surface of steel slag particles. It forms certain chemisorption with O and other elements in the geopolymers which resulting in physical interaction between irregular steel slag particles and the geopolymer matrix. However, further increase of EAFS to 30 wt.% lower the bonding strength between the EAFS and metakaolin geopolymer. This is due to the agglomeration between small particles of EAFS which lower their dispersion in the geopolymer [15].

Furlani et al. (2018) have confirmed that the addition of 40 wt.% EAFS to 60 wt.% of metakaolin yield highest compressive strength and specific surface area. However, further increment of EAFS content will deteriorate the mechanical properties [16]. The improvement of compressive strength is related to the presence of calcium and magnesium oxides in the slag leading to the formation of stronger mechanical bonds replacing part of the original N-A-S-H based reference material. Moreover, the steel slag strongly reduces the presence of cracks in the geopolymers due to the presence of inert materials, affecting shrinkage during hardening. However, the lower amount of reactive materials influence the mechanical performance [16]. Although highest compressive strength is expected to be achieved at 100% of EAFS incorporation, the strength starts to deteriorate when the content is more than 60 wt.%. This is due to the presence of unreacted inert material in the EAFS which decreases the number of chemical bonds that is formed during geopolymerization [16]. Hoy et al. (2018) utilizes steel slag geopolymer binder in Recycled Asphalt Pavement (RAP). The content of SS in the RAP-S geopolymer is varied into 10% and 20%. It is observed

that the unconfined compressive strength (UCS) value of geopolymer increases with increasing EAFS [18]. The compressive strength of pure EAFS geopolymer reported by Češnovar et al. (2019) is 39.66 MPa [19].

2.3 Ladle Furnace Slag (LFS)

The utilization of LFS in geopolymer is very scarce. Some researches available on LFS in geopolymer are the usage as geopolymer binder and geopolymer paste [20,21]. Wang et al. (2017) have discussed on optimizing the factors affecting the properties of geopolymer binder [22]. Adesanya et al. (2017) have studied on effects of alkali and silica contents on mechanical properties, water absorption, and porosity of the produced geopolymer samples [21]. Češnovar et al. (2019) have reported on the effect of curing parameter on the mechanical properties of LFS [19].

FTIR analysis of LFS are quite similar to EAFS, major bands of LFS also lies in the 970-1160 cm^{-1} range which is due to the symmetric stretching vibration of Si-O-Si and the Si-O-Al bonds [19]. The intensive bands at 1429 cm^{-1} is related to the CO_3 in CaCO_3 . This bands also exist at 874 cm^{-1} . The vibration bands at 1648 cm^{-1} in activated samples are due to H-O-H bending vibrations, indicating the presence of water in the hydration product, which are not present in the starting material. The peaks around 1030-1101 cm^{-1} correspond to the asymmetric stretching vibration of the Si-O bonds. The spectra show the shift in position of the stretched vibration bands of the Si-O bonds from 1030 cm^{-1} in the slag to 1101 cm^{-1} in the activated slag. The increase in wavenumber and the broadness of the bands with the increasing silica contents in the activated slag are linked to the formation of a polymerized Si-O network, with amorphous structure. Also, the spectra indicate a low-intensity absorption band at around 600 cm^{-1} which has been associated with the vibration of T-O₄ groups (T represents tetrahedral Si or Al). The absorbance peaks at 1648 cm^{-1} and a broad peak around 1071 cm^{-1} exhibit great resemblance with a mixture of C-S-H and N-A-S-H gels. The remaining wide absorption bands at the 1406-1482 cm^{-1} region corresponds to asymmetric stretching vibrations of O-C-O bonds of carbonates group, which suggests that carbonation of the sample have occurred during the long curing period, since carbonate phases are not observed after 28 days of curing [21].

γ -dicalcium silicate and mayenite are identified as the major minerals from XRD analysis of LFS. There is an increase in the amorphous phase with a weak amorphous “halo” with 2θ values ranging from 23 to 38° as compared to the raw slag. This implies that some constituents of the LFS have dissolved in the alkaline solution, leading to the formation of an amorphous hydration product, likely that of C-(N)-A-S-H gel. The C-(N)-A-S-H gel is a likely reaction product even though the amorphous halo is overlapped by the peaks of dicalcium silicate and mayenite hydrate. Furthermore, this development in the evolution of phases of the samples shows the potential of crystalline slag reactivity in alkaline solution. The considerable factor is the existence of mayenite as one of the main minerals; this calcium aluminate mineral is known to react rapidly. Also, it should be noted that some peaks in the patterns are not identifiable, notably at 2θ values of 48.5° and 60° [21].

Unreacted slag grains embedment in geopolymer matrix were observed by Adesanya et al. (2017) from SEM micrograph of the polished surface of LFS geopolymer with some microcracks were also clearly exhibited in the microstructure. Moreover, a narrow reaction ring can be observed at the interface between the unreacted grains and the alkaline activated paste [21]. According to Adesanya et al. (2017), LFS is commonly regarded as having limited reaction with alkaline. Although usually being used as alkaline activated slag with metakaolin, it also shows potential to be used as sole precursor for alkaline activated binders [21]. The compressive strength of the optimized geopolymer paste specimen was 65 MPa at 28 days. Wang et al. (2016) reported that the curing of LFS geopolymer in saturated limewater shows increasing compressive strength with increasing age [20]. It is reported by Češnovar et al. (2019) that the compressive strength of LFS as sole aluminosilicate source is about 49.55 MPa [19].

3. FACTORS AFFECTING PROPERTIES OF SLAG-BASED GEOPOLYMERS

Generally, the alkaline concentration, type of alkaline medium, curing temperature, curing condition and weight ratio played important roles in geopolymer formation generally. However, in case of slag-based geopolymer, the effect may differ depends on the source or type of slag itself.

Alkaline treatment are needed in the process in order to yield the molecular units of the geopolymer [5]. The transformation starts with the dissolution of raw material in highly alkaline solution which releases Si and Al species creating supersaturated solution of SiO_2 and Al_2O_3 species. By doing so, the process will progress to form coagulated structures, gelation and finally hardening through the formation of three-dimensional geopolymer matrix [4,12]. Moreover, the nature and dosage of the alkaline determine the final properties of geopolymers [6]. Thus, it is very

important to neatly control the alkaline treatment parameters as it is largely affecting the final properties and cannot be altered once the process have been started since geopolymerization happens in a very short period of time.

Therefore, alkaline concentration has been actively investigated on the effect on geopolymerization. Previous study have revealed that the optimum concentration for alkaline solution is 6-8 M; further increase to 10 M tend to reduce the compressive strength of the geopolymer [14]. The increase in compressive strength can also be observed in LFS when alkali concentration is increased causing the pH to increase and accelerate the destroying of LFS resulting in a more compact colloidal structure [20]. On the contrary, too high alkalinity causes the crystalline deposits on the geopolymer surface by excess alkaline metals (K^+ and Na^+) in the paste. This causes the precipitate in the binder gel (C-A-S-H) in the early stage of hydration, deterring further formation of binder phases and thereby decreasing strength. The compressive strength for Na content of 4.3 after 28 days shows 47 MPa while the lowest is for Na content of 6.3 with 38 MPa [21].

Next, the type of alkaline activator is confirmed to be affecting the compressive strength of geopolymer [5,19,20]. The common type of alkaline being used are the mixture of hydroxides (NaOH or KOH) and their silicates counterparts (Na_2SiO_3 or K_2SiO_3) [23]. It has been identified that larger sized cation (K^+) tend to form larger silicate oligomers which often preferred by Al (OH) to bind. Thus, activator containing KOH will display better setting time and stronger compressive strength than that of NaOH because KOH solutions comprises of more geopolymer precursors [14]. It is a fact that the mix of both alkali hydroxide and alkali silicate yield better compressive strength such that it is found that mixing sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) have contributed to better compressive strength at elevated temperature as compared to sodium hydroxide as single activator [6]. This is also have been observed in geopolymer concrete when using both alkaline solution as alkaline activator due to the formation of higher amount of calcium silicate hydrate (CSH) when sodium silicate is used [24]. Meanwhile, it is asserted that the addition of sodium silicate prompted the higher compressive strength of BFS based-geopolymer by improving the dissolution rate of Si and Al as well as taking advantage of weaker Al-O as compared to Si-O bonds that causes Al to dissolve rapidly in alkaline solution [14]. The sodium silicate acted as alkaline activator, plasticizer, and binder. Meanwhile, the alkaline hydroxide used for dissolution of aluminosilicates sources. In addition, alkaline solutions promote a certain quantity Si and Al atoms to dissolve the aluminosilicates sources, form monomers in solutions, and then polycondense to form rigid network [25]. Despite the alkaline activator being a significant factor, it is important to note that strength development may differ for slags of different origins [6].

The ratio of sodium silicate to sodium hydroxide is important, as it is worthy to note that the higher Na_2SiO_3 content has improved the compressive strength of the geopolymer. However, the NaOH/ Na_2SiO_3 ratio more than 60:40 for example of 50:50 will exhibit reduction in its strength [18]. The same effect can also be achieved by increasing the silica content in the raw material which show an increase in compressive strength until it reach 20% of SiO_2 content which then maintained constant values [21].

Curing is crucial in geopolymerization because hardening, or setting, or geopolymerization, occurs at low temperature, below 100 °C, or at room temperature [5]. It is well-known that modification of aluminosilicate materials, calcium content, alkali content, alkali type and also water content affect the setting time of geopolymer [11]. According to study conducted by Bai et al. (2018), with the incorporation of slag, the curing time could be accelerated by 100% as compared to geopolymer without slag [15]. This is because, during curing process, the double charged calcium ions are introduced by slag combines with hydroxyl to form hydroxide precipitates. Then, large solid particle with lower surface energy of silicate polymers is formed due to the pH reduction around the hydroxide precipitates. Hence, the increased rate of hardening can be observed as the nucleation and polymerisation between the soluble silicate and solid particles were improved. Moreover, the blending of slag not only changes setting characteristics but also significantly alters the mechanical properties of the resulting geopolymer [11]. Furthermore, correct curing method and curing time helps to improve the mechanical properties of geopolymer. Bending strength is asserted to be most affected by the curing method applied for geopolymer [15]. However, in most cases the curing temperature and curing environment are the most researched by previous studies due to their strong effect on the other mechanical properties of geopolymers.

Calcium-rich precursors such as slag will exhibit obvious effect upon curing temperature changes. Depolymerization and polycondensation occur at room temperature as well as under high temperature, it is concluded that as curing temperature increases, the compressive strength does increased [22].

Moreover, the curing of LFS specimen in saturated limewater can greatly improve the engineering properties of the geopolymer compared to air curing. Curing in saturated limewater promotes the polymerization reaction due to the incorporation of additional alkali; thus, the engineering properties of LFS cured in saturated limewater outperform those of LFS cured in air and shrinkage is effectively prevented [20].

4. CONCLUSION

These findings enhanced our understanding of the role of slag in geopolymer which may be different for different types of metallurgical slag. However, the properties of different slag may be good for certain types of application. The reaction mechanism although the same, depends on a few factors such as alkali concentration, modulus, type of alkaline activator, curing temperature and many more.

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