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Synthesis and Characterizations of High Carbon Ferrochrome (HCFC) Slag Based Geopolymer

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Abstract

In the present Investigation, Geopolymer (GP) is made using High carbon Ferrochrome (HCFC) slag by synthesizing silicon and aluminum present in it with alkali liquid solution which binds other non-reactive materials present in the slag. The compressive strength of GP is found to be 11 MPa after 7-days of air curing. Increasing air curing time to 28-days, the strength is measured to be 15 MPa. XRD analyses indicate that there is gradual transformation of crystalline phases to non-crystalline glassy phases. SEM images show presence of more amount of glassy phase after air curing for a longer time. This is also corroborated with mechanical properties such as compressive strength. TGA results are also discussed for both uncured and cured GP samples. DSC isotherms indicate oozing out of inbuilt water present in the prepared GP materials which is an indication of condensation polymerization reaction occurring during the formation of Geopolymers.

Keywords: Geopolymer, Industrial waste, Slag, Cement, alkaline activator, phases, Absorption band Morphology, Compressive strength, Condensation polymerization

1. Introduction

Portland cement (PC) is a common structural material, which is widely used in construction of structure [1–4]. PC production causes environmental problems due to release of carbon dioxide (CO_2) [5]. The production of PC is extremely energy intensive process [6]. Therefore, research community have used industrial wastes such as fly ash (FA), pond ash (PA), slag, bottom ash (BA), quarry dust (QD), etc. as resource materials for the production of Geopolymer [1–5]. It is no wonder why researches are being carried out for the replacement of PC with new generation material, *i.e.*, GP. Therefore, GP technology has enabled to produce an alternative material to replace PC [7, 8].

One of the wastes is slag which is obtained from Ferro-alloy industries during the production of ferrochrome [1–5]. The slag contains variety of oxides such as magnesium oxide, iron oxide and chromium oxide which exist in more than one oxidation states. Also, Ferrochrome slag may contain elemental chromium [7, 8]. The major constituents of the slag are Cr_2O_3 , FeO , SiO_2 , MgO , Al_2O_3 , CaO [7, 8]. In the year 2008, Davidovits has described GP to be a future constructional material and is predicted to replace PC in constructional areas [9, 10]. Reports are available

which indicate waste materials *i.e.*, Fly Ash, Pond Ash, Bottom Ash, slag, etc. are suitable for production of Geopolymer [7, 8]. These oxides form chain-like structure by the reaction with active alkali solution such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) in combination with sodium silicate [7, 8]. Thus, Geopolymer can be considered as an innovative constructional material for 21 century [11, 12]. Many researchers have described Geopolymer structure [7, 8]. The advantages of using GP materials lie in a fact that they are cheaper, unhazardous, fire resistant and environmental friendly [7, 8].

The corrosion test and p^H measurements have revealed that GP material has low leachability, excellent frictional resistance. Therefore, it can be used for construction of roads [13–16].

In the current work, HCFC slag is chosen from a ferrochrome industry as main component for preparing Geopolymer. Attempt is also made to characterize both HCFC slag and as-prepared Geopolymer material through different technique such as mechanical, structural, morphological, and thermal analyses.

2. Experimental details

2.1 Chemicals and materials

During the manufacture of Iron and Chrome alloy, slags are produced in smelter by the reaction of fluxing agents such as quartzite, bauxite, dolomite, corundum, lime and olivine with gangue materials of the ore. Usually, the foam slag in the smelting furnace contains 1.0–6.0 Cr depending on the charge at the reaction temperature.

In the present investigation, a HCFC slag from ferrochrome industry (**Shyam ferroalloys** Durgapur, West Bengal, India) is collected and composition is shown in **Table 1**. Other ingredients used are sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3), which are procured from M/S Loba Chemicals, India and Merck, India, respectively. These reagents are used for treating HCFC slag. NaOH acts as an activator and Na_2SiO_3 act as a polymerization agent. Water soluble plasticizer (SiKA) is used in the processing of Geopolymer which acts as a plasticizers *i.e.*, an agent for enhancing plasticity of the product (Geopolymer). The SiKA chemical is procured from a reputed firm located in the southern parts of India (Visakhapatnam market). All relevant details such as image, color, density, supplier name of raw materials used for preparing GP are described in **Table 2**.

2.1.1 Alkaline liquid

A combination of sodium silicate solution and sodium hydroxide solution are chosen as the alkaline liquid because they are cheaper [17–20].

Constituents of HCFC slag with percentages	SiO ₂ = 28–31
	MgO = 26.0–26.5
	Al ₂ O ₃ = 23.5–24.5
	CaO = 7.5–8.5
	Cr ₂ O ₃ = 4.5–5.4
	FeO = 2.5–3.0
Sources	Syam Ferroalloys, Durgapur

Table 1.
Chemical composition (%) of HCFC slag (XRF analysis).

Chemicals	Sample Images	Color	Density (g/mL)	Supplier
HCFC Slag		Blackish Gray	2.84 (Specific Gravity)	Shyam ferro alloys
Sodium Silicate		Brown Color	2.4 g/cm ³	Merck India
Sodium Hydroxide		Color less	2.13 g/cm ³	Merck India
Sika		Grayish	~1.18 kg/L (Relative density)	Vizag market

Table 2.
Ingredients sample image, color, density, supplier of HCFC slag.

2.1.2 Sodium hydroxide (NaOH)

The laboratory grade sodium hydroxide (NaOH) solid is taken in the form of pallets (3 mm, diameter), with a specific gravity of 2.130, 98% purity. NaOH solution is prepared by dissolving the pellets in distilled water. NaOH solution of 8 M concentration ($8 \times 40 = 320$ grams of NaOH solids per liter of the solution, where 40 is the molecular weight of NaOH) is used for preparing geopolymer. Similarly, NaOH solutions of other concentrations are prepared as discussed above.

2.1.3 Sodium silicate (Na_2SiO_3)

Usually, sodium silicate is common name of compounds which is expressed by a formula $(\text{Na}_2\text{SiO}_2)_n\text{O}$. It is a semi-viscous solution having sp. gravity of 2.4 g/cm³. The open chemical structure of sodium silicate is shown in **Figure 1**.

2.1.4 Plasticizer

In order to improve plasticity of the geopolymer, a SiKA compound is used along with reacting materials during processing of geopolymer. It is a low cost material. In addition, extra water (5–6 mL) is added to SiKA plasticizer solution. In designing

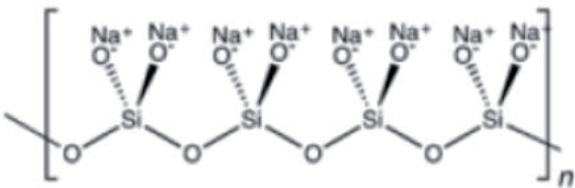


Figure 1.
Chemical structure of sodium silicate [Wikipedia.org/wiki/Sodium_metasilicate].

the mixtures the parameter “water-to-geopolymer solids ratio” by mass is considered to be important. Thus, the total mass of water is the sum of the mass of water contained in the sodium silicate solution, sodium hydroxide solution, and the mass of extra water [21, 22].

3. Synthesis procedure

Steps for geopolymer preparation with high carbon ferrochrome (HCFC) slag are described below (Table 3) [23, 24].

3.1 Raw material preparation

HCFC slag is collected from Shyam ferroalloys Durgapur, West Bengal, India in the form of lumps. The lumps are crushed and ground in a ball mill, followed by sieving through mesh (size 240). The fine particles, thus obtained, are dried in an oven for 2 hrs at 120°C.

The alkaline activator used for preparation of Geopolymer (GP) is a combination of sodium silicate and sodium hydroxide in appropriate proportion. While preparing the solution mixture in a flask, it is kept on a hot plate with a magnetic stirrer. The alkaline liquid is prepared by mixing sodium silicate solution and sodium hydroxide solution. The solution is continuously stirred with a magnetic stirrer.

Plasticizer (SiKA) with extra water is poured to the liquid mixtures.

3.2 Geopolymer preparation with raw materials

Prepared HCFC slag is poured into a mixture container. Then, alkali liquid solution is added slowly to the slag and is manually mixed. The mixture is left five minutes for polymerization reaction. Different mixture compositions are prepared.

3.3 Molding, casting and compaction

Geopolymer prepared with different variables are designated as S1, S2, and S3 (Table 2). The shape of mould is cylindrical 70 mm height and 55 mm diameter, respectively. The poured materials are rammed for compaction. The whole mixture is allowed to set for 3 minutes before demoulding samples. These are then rammed or struck for compaction followed by setting for 3 minutes before demoulding the sample. GP samples are removed from molds and are used for further investigations. It is to be mentioned their inside surface are coated with diesel oil to ensure smoother surfaces of samples. The entire process has been described (Figure 2).

Sample code	HCFC slag	Sod. Silicate (SS)	Alkali (8 M, NaOH) (SH)	Water soluble Plasticizer (Sika)
S1	85%	12%	3%	1-2 ml
S2	85%	13%	2%	1-2 ml
S3	85%	14%	1%	1-2 ml

Table 3. Variation of SS and SH with other parameters as curing time 24 h, curing temperature 70 C, mesh 240 [24].

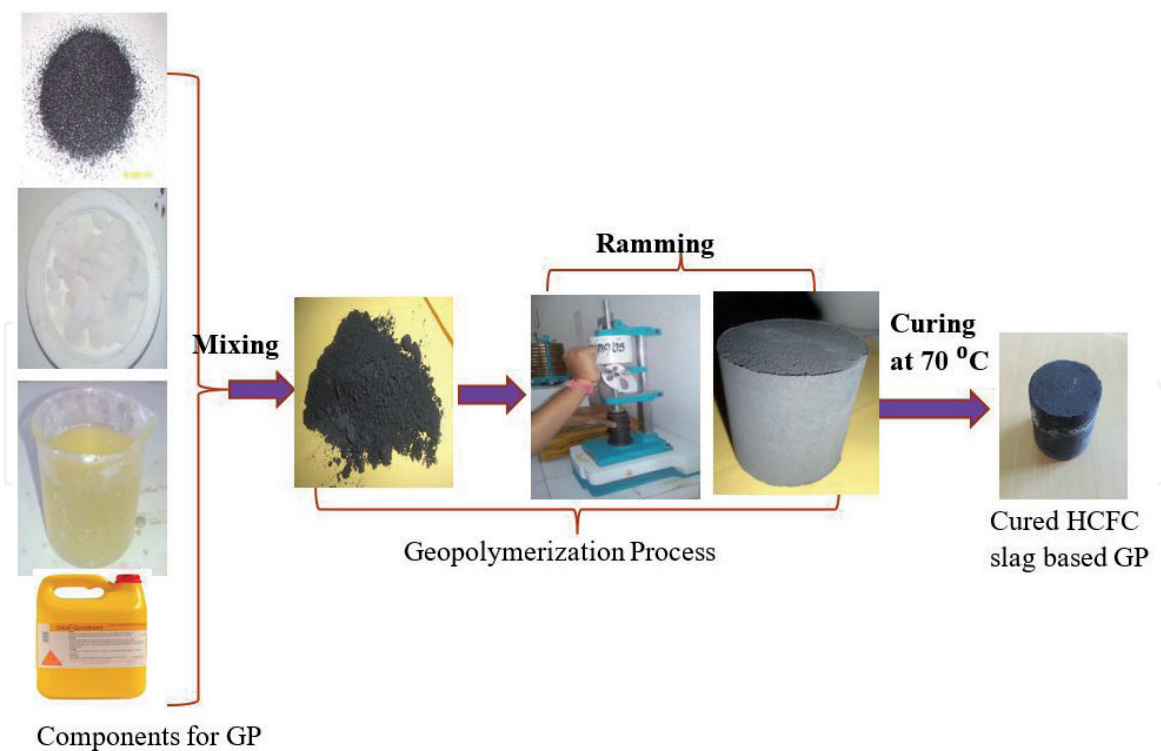


Figure 2.
Schematic diagram of process for preparation HCFC slag based GP product [23].

3.4 Curing

The green samples are kept for heating in an oven for 24 h at 70°C followed by curing in open air (7-days and 28-days) [25, 26].

Specimen(s) are used in different characterizations such as XRD, FTIR TGA, DSC, etc.

4. Characterization techniques

Different phases in the HCFC slag are identified using X-ray diffraction technique. Essentially, the phases are determined by qualitative analysis of the phases through JCPD file. For this purpose, ferrochrome slags powdered are dried 110°C for 24 h and crushed into fine powdered use in the examination. For analysis purpose a Phillips PW-1710 Advance wide-angle X-ray diffractometer and Phillips PW-1729 X-ray generator using CuK α radiation (wavelength, $\lambda = 0.154$ nm). The generator is operated at 40 kV and 20 mA. The powder samples are placed on a quartz sample holder at room temperature and are scanned at diffraction angle 2θ from 10° to 60° at the scanning rate of 5°/min. X-rays diffraction studies are made with treated GP samples at different stages of curing. Similar analyses are also made with the as received HCFC slag for comparison purpose.

FTIR technique is used to analyze the presence of functional groups, formation of chemical linkage using a FTIR spectrometer (Nexus-870, Thermo Nicolet spectrophotometer) in range of 400–4000 cm^{-1} . The instrument parameters are kept constant (50 scan at 4 cm^{-1} resolution, absorbance mode). In this spectrometer, the IR radiations from an IR source are passed through the sample and the amount of energy adsorbed is recorded by suitable detector and is guided through an interferometer where a Fourier Transform is performed on the output signal. The pellet (13 mm diameter, 0.3 mm thick) so prepared is used for IR characterization. Before

running the samples, a background spectrum is collected. Then pellets samples are put in a sample holder. The pellets are exposed to IR radiation in the spectrometer and data were collected.

Surface morphologies of prepared materials are analyzed by electron microscope (SEM using Carl Zeiss Supra 40). SEM is a microscope that uses electrons in place of light to produce image. In this measurement, the electron beam produced from electron gun is focused on a small portion of the sample that is kept in vacuum. Detector collects the output signals during the interaction of electrons with the sample and that is sent to a computer. This forms the final image. Special preparation technique is needed for the sample to avoid moisture absorption. All non-conducting materials need thin layer of conducting coating. This is done by 'sputter coater'. Operating voltage was 4 kV. The sample is placed in a small vacuum chamber. The argon gas is ionized in the applied electric field to form argon ion (Ar^+). The argon ions knock gold atoms from the surface of the gold foil and get deposited on sample. The elemental analyses are carried out with the help of EDX attached to the microscope.

TGA analysis is a type of thermal analysis that measures mass change of materials with change of temperature. The purpose of doing such experiment is to measure volatile content, thermal stability, degradation characteristics, etc. The as-prepared fine powdered of HCFC slag for Geopolymer preparation and as-prepared cured Geopolymer materials are also run in a thermogravimetric (TG) analyzer Perkin Elmer Pyris Diamond analyzer. Heating rate is taken $10^\circ\text{C}/\text{min}$ during the experiments and is performed in an inert atmosphere (N_2 gas). The TGA run is carried out in the temperature range of $50\text{--}300^\circ\text{C}$.

Differential scanning calorimetry (DSC) is a technique for measuring the energy necessary to establish a nearly zero temperature difference between a substance and an inert reference material, as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate. The technique provides qualitative and quantitative information about physical and chemical changes that involve endothermic or exothermic processes or changes in heat capacity using minimal amounts of sample. It has many advantages including fast analysis time, typically thirty minutes, easy sample preparation, applicability to both liquids and solids, a wide range of temperature applicability and excellent quantitative capability.

DSC has been used in the evaluation of small transitions such as multiple phase transitions in liquid crystals and those due to side chains in polymers which cannot be resolved by most other techniques. It allows accurate determination of temperatures associated with thermal events. Temperature can be calibrated with respect to one or more standards which allow highly accurate, precise and reproducible values. The technique reveals the thermal history imparted to thermoplastics as a result of different processing conditions. The information generated can be used to vary heating rates to deliver the required degree of crystallinity. Differential scanning calorimetry (DSC, Perkin Elmer Pyris Diamond) was used for determination of crystallization. Samples (5–10 mg) were placed in sealed aluminum pans and scanned under a constant nitrogen purge ($20\text{ mL}/\text{min}$). Subsequently, the samples were heated from $50\text{--}300^\circ\text{C}$ at a rate of $10^\circ\text{C}/\text{min}$, cooled to 30°C at the same rate and held for 2 min to stabilize. Finally a second scan was carried out from room temp to 300°C . The result of enthalpy was noted from the second scan. DSC study is made for measurement of enthalpy of the two materials *i.e.*, as-received HCFC slag and GP prepared with HCFC slag. The test is carried out in nitrogen environment (N_2).

Tests to obtain the stress–strain curves prepared HCFC slag based GP in compression are performed using an Ultimate Tensile machine (UTM), AIMIL

COMPTTEST 2000 model and is determined by standard ASTM which is inbuilt in the UTM machine. The tests on 100 × 200 mm GP cylinders are performed by using the displacement-control mode available in the test machine. It took approximately 30 to 60 minutes to complete each test in order to obtain the stress–strain curve(s). Loading in compression over a period is found to cause reduction in the measured value of the compressive strength of test cylinders. Maximum load is taken at the point of fracture of the sample. The specimen(s) is demoulded compression testing.

5. Results and discussion

Ferrochrome slag is obtained as waste material during production of ferrochrome alloy in a smelter. Pouring temperature of Slag is around 1500°C. The slag contains oxides of magnesium, iron and chromium in their different oxidation states. Chromium present in the slag may either be trivalent (+3 oxidation state) and or hexavalent (+6 oxidation state). Some elemental chromium may also be present in the slag. The oxides are usually spinel phase (*i.e.* FeCr₂O₄). MgAl₂O₄ spinel phase is also there in the slag. Presences of these spinel phases help the slag to enhance mechanical, chemical and thermal properties both at ambient and elevated temperatures [27, 28]. **Table 1** depicts chemical analysis of ferrochrome slag. It is evident that SiO₂, Al₂O₃, Fe₂O₃, and Cr₂O₃ are present in major quantities, while CaO, MgO, FeO are present in a minor quantities [29]. The ground and sieved (240 meshes) of as-received slag is dried for 2 h at 120°C for removal of moisture. The sieved HCFC slag is ready for Geopolymer preparation.

Geopolymer samples are made by treating with different molar concentration of sodium hydroxide (NaOH). Effect of different molar concentration of alkali materials on the compressive strength is studied. The strength properties of as-prepared Geopolymers are shown in **Figure 3**. S1, S2, S3, S4 represent Geopolymer prepared with 6 M, 8 M, 10 M, and 12 M solution of NaOH, respectively. These are cured at 70°C in an oven, followed by 7-days and 24-days of curing in air. It is well known that sodium plays an important role for polymerization of alumina (Al₂O₃) and silica (SiO₂), which is present in the slag. Each pair of bar drawn in **Figure 3** is

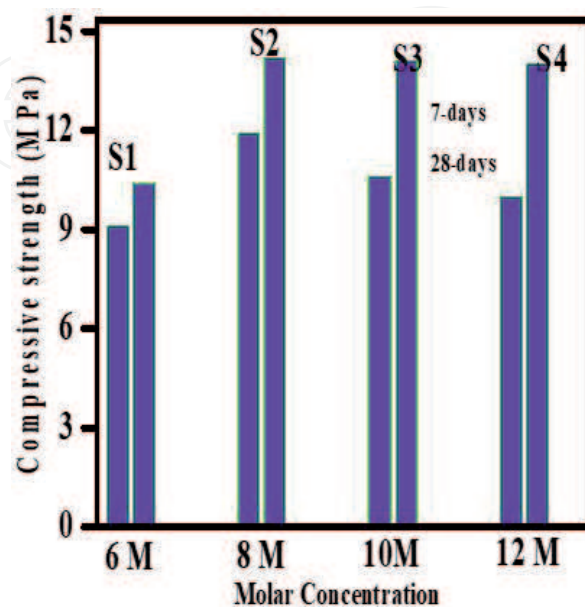


Figure 3. Compressive strength vs. molar concentration at 7-days and 28-days air curing of HCFC slag based GP samples prepared by optimized parameters [23].

showing data on comparative strength scale for a Geopolymer prepared with different molar concentration, cured for 7 days and 28 days, respectively. It is found that maximum compressive strength has been achieved for the Geopolymer prepared with 8 M NaOH concentration [30–33].

Figure 4A and B show superimposed XRD pattern obtained from two different types of samples i.e., prepared with as-received HCFC slag (**Figure 4A**) and geopolymer prepared with optimal treatment combination (**Figure 4B**). Sharp XRD peaks can be seen in XRD pattern of as received slag (**Figure 4A**), whereas broaden peaks can be seen in geopolymer prepared with optimal treatment combination. It can infer that the slag material has crystalline phases and geopolymer has non-crystalline glassy phases. Sharp peaks in the as received material are identified to be quartzite and mullite phases, whereas broaden line profile of XRD pattern of geopolymer are glassy phases formed chemical and thermal treatment.

The morphology of HCFC slag and GP samples prepared with optimal treatment combination is studied (**Figure 5A and B**). **Figure 5A** shows SEM micrograph of HCFC slag which consists of fused mass of oxides with some glassy phase spread over the matrix. Since the slag material is poured from a high temp to a room temperature, therefore, faster cooling rate results. **Figure 5B** is a magnified image of Geopolymer as shown in the earlier slide. The picture shows morphology of Geopolymer prepared with optimal treatment combination. At lower magnification i.e., 500 KX, it shows crystalline needle shape phase oriented in a random direction (**Figure 6B**). Additionally, there is some region of glassy phase co-exist together with the crystalline phase. The sample, therefore, is further investigated at a higher magnification i.e., 1500 KX (**Figure 5B**). **Figure 5C** shows the bundles of rod-shaped phase oriented in the same direction. This crystalline phase has occurred due to cross-linking of alumino-silicate Geopolymer phase by reacting with the sodium of sodium hydroxide [34]. EDS analyses are done during the SEM studies (**Figure 7**). The elements such as Na, O, Al, Ca, Mg, Cr are present both in crystalline and glassy phases of as-prepared HCFC slag based GP (**Figure 7B**). However, all the elements present in GP are found in as-received slag except element such as sodium (**Figure 7A**). Occurrence of element sodium in GP is due to the reaction sodium compound with slag during polymerization process.

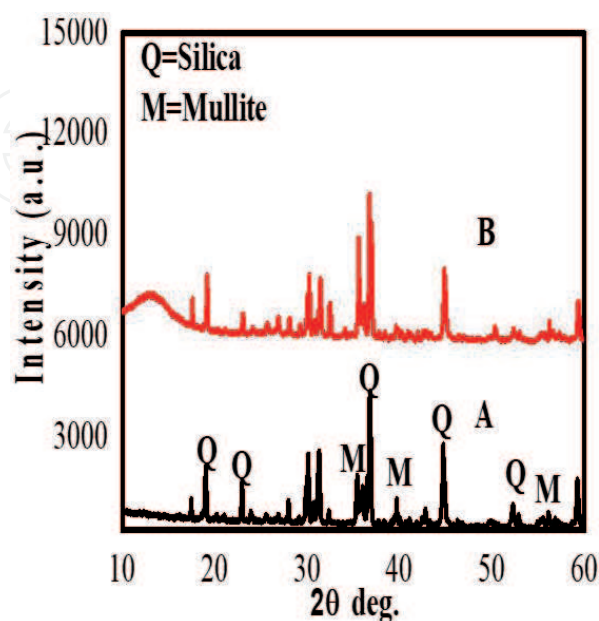


Figure 4.

XRD pattern of grinded with sieved as received HCFC slag (A) and as-prepared HCFC slag GP prepared by optimized parameters [23].

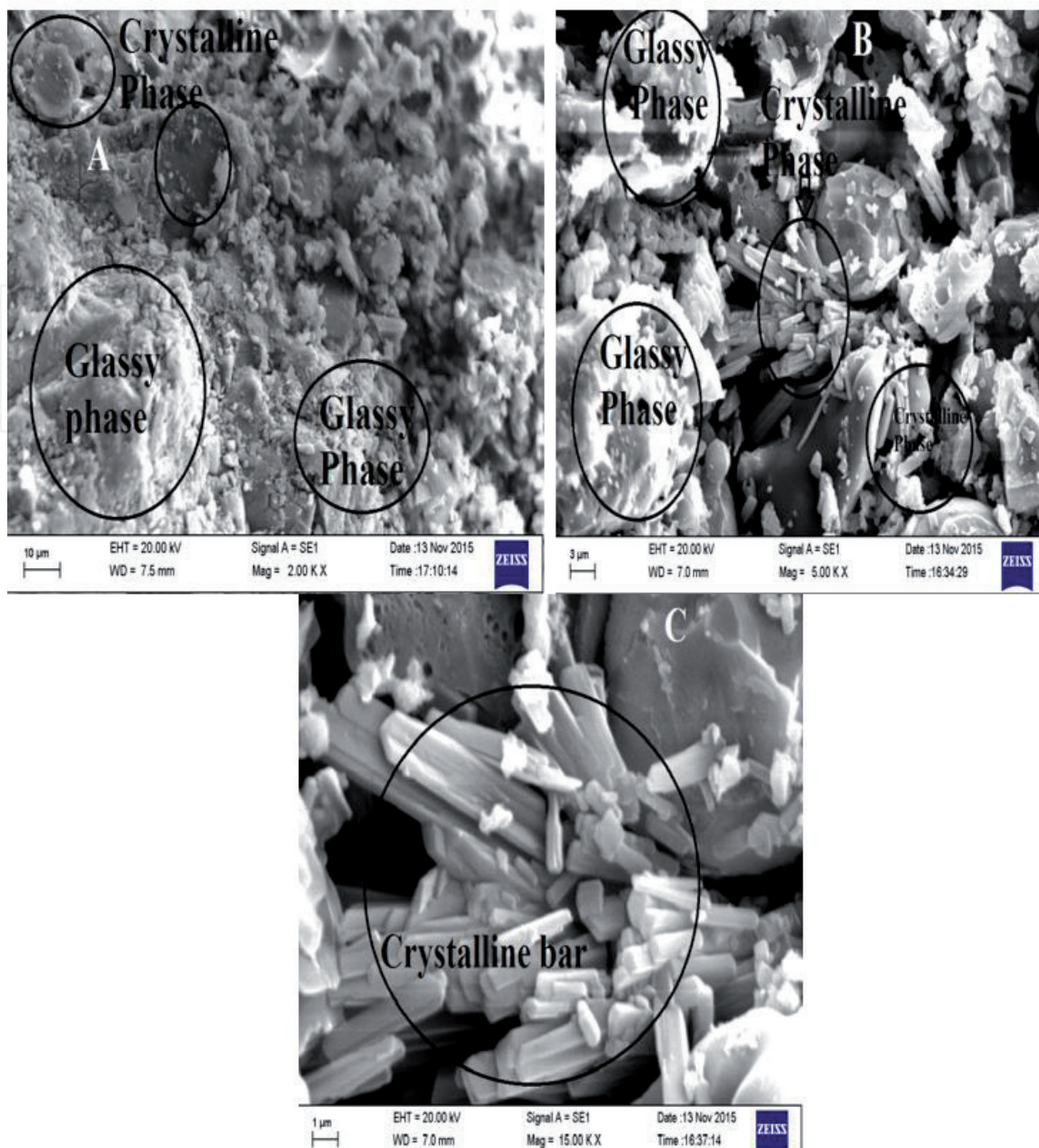


Figure 5. SEM images of grinded with sieved of as-received HCFC slag (A), slag based GP samples prepared by optimized parameters [23].

FTIR spectra of the slag and GP material are shown in **Figure 6**. FTIR spectra indicate the presence of different absorption bands in each category of material. **Figure 6A** indicates the presence of different absorption bands occurring at different wave numbers *i.e.*, 3441, 2918, 1638, 1442, and 887 cm^{-1} . Bands are in agreement with stretching vibrations of O-H bonds (3441 cm^{-1} wave number) and H-O-H bending vibrations (1638 cm^{-1} wave number) of interlayer adsorbed H_2O molecule [35]. The hydroxyl-stretching band of water plays an important role and peak shift of the FTIR spectra is significant. Absorption band ensue at 887 cm^{-1} wave number is attributed to Si-O band and signifies the occurrence of silicate groups. Presences of $\text{Al}^{3+}\text{O}^{2-}$ absorption bands are also indicated near 805 cm^{-1} wave number [35]. Stretching bands is found at 440 cm^{-1} wave number and signifies the occurrence of Fe-O band [36]. However, the absorption bands are not found to occur in as-received HCFC slag. This is due to the formation of Geopolymer after treating and curing of as-received HCFC slag.

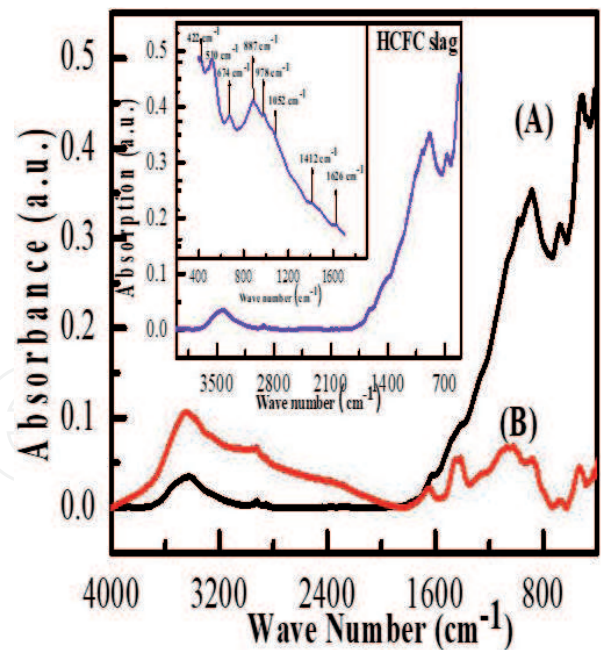


Figure 6. FTIR spectra of grinded with sieved as-received HCFC slag (A) and HCFC slag based GP prepared from optimized parameters (B) [23].

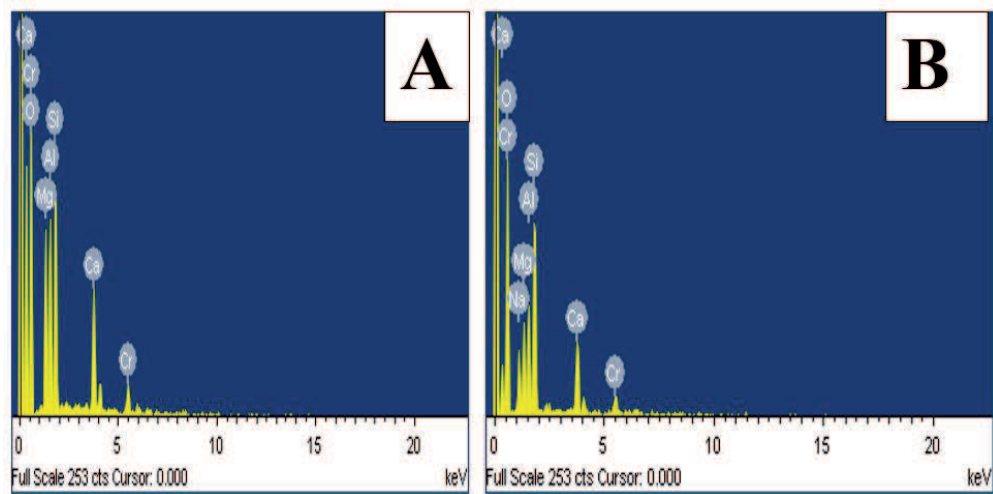


Figure 7. EDS of grinded with sieved of as-received HCFC slag (A) and slag based GP samples prepared (B) by optimized parameters [23].

In TGA analysis, the weight loss is estimated for different temperature, ranging between 50 and 800°C and data are plotted as shown in **Figure 8**. TGA runs are taken from two samples, HCFC slag and GP. There is a sharp decrease in weight percentage beyond 100°C. This is attributed to the loss of water molecules. Between 100 to 350°C, there is a rapid declination the curve. Beyond 350°C, a little change is observed. The average total loss in weight (%) increases and decreases within the range of temperature 350–700°C [37, 38].

Differential scanning calorimetry (DSC) studies are made for both the samples i.e. HCFC slag and the GP formed it. It may be observed that there is no change in heat flow for slag (**Figure 9A**) whereas; there is significant change in heat flow for GP made from slag (**Figure 9B**) at 141°C. This is due to expulsion of physically bound water within the GP materials [39].

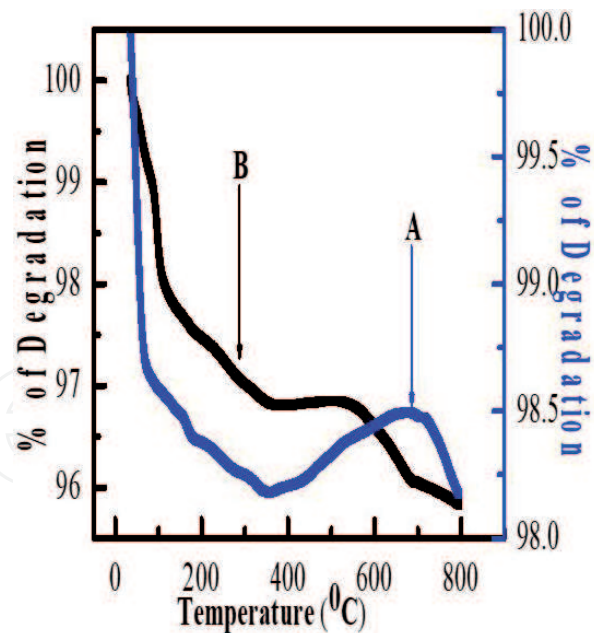


Figure 8.
TGA curve of grinded with sieved as-received HCFC slag (A) and HCFC slag based GP prepared with optimized parameters (B) [23].

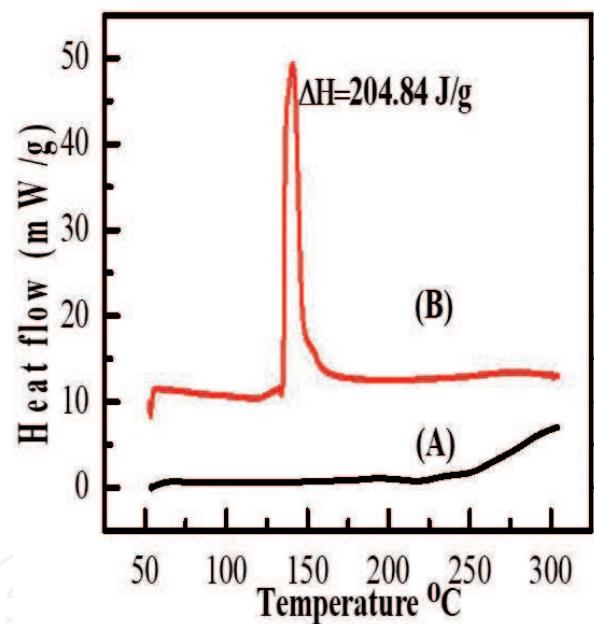


Figure 9.
DSC isotherm of grinded with sieved as-received HCFC slag (A) and HCFC slag based GP prepared with optimized parameters [23].

6. Conclusions

Geopolymers are prepared from HCFC slag successfully by treating with the alkali materials. The structure and morphology of Geopolymer are studied using different characterization technique. The microstructure of Geopolymer shows needle-shaped, randomly oriented crystalline phase, embedded in glassy phases. Together with microstructural observation, SEM/EDS analyses show presence of alkali materials in the Geopolymer which indicates occurrence of reaction of the HCFC slag with alkali solution during the Geopolymerization process. The

maximum compressive strength is obtained to be 15 MPa by curing at 70°C in an oven for 24 h followed by cooling for 28-days in air. XRD pattern has clearly shown conversion of crystalline phase present in the slag has been transformed to glassy phase during the formation of Geopolymer. There is a marked difference observed in the FTIR spectrums. The numbers of peaks of as-prepared samples are much more in comparison to peaks present in as-received samples. This is due to formation of new bonds within the phases. TGA curves have revealed, for both the materials there is a gradual degradation material in the temperature range studied. DSC results show that there is a exothermic peak in Geopolymer at 150°C. This is due to elimination of water during condensation polymerization process occurring in the geopolymer.

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