


Article

Impact of Synthetic Parameters on the Compressive Strength of Bagasse Ash-Clay Geopolymer

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Abstract: The impact of different parameters on the compressive strength of geopolymer synthesized from clay and bagasse bottom ash is reported. Geopolymer was synthesized from thermally activated clay and bottom bagasse ash using sodium silicate and sodium hydroxide as activator. The maximum dissolution of alumina and silica from the bagasse ash and clay maintaining different alkali conditions is studied. The resulting geopolymer synthesized under different conditions is studied for compressive strength. Different characterizations of the resulting geopolymer were carried out using different analytical instruments. The results indicated that the dissolution and strength of geopolymer have close relationships with the alkali concentration, solution to solid ratio and curing period. The highest compressive strength of 25 MPa was observed for 8M NaOH, 24 MPa for 0.3 solution to solid ratio, 30 MPa for 60% clay and 30 MPa for 27 days of compressive strength.

Keywords: bottom bagasse ash; activated clay; geopolymer; compressive strength; curing period; alkali concentration



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1. Introduction

Geopolymers are three dimensional inorganic polymers with amorphous and some semi amorphous phases, having Si-O-Al linkage synthesized for the first time by Joseph Davidovits 1982. Geopolymers comprise a tetrahedral coordinate structure of Si⁴⁺ and Al³⁺ ions with oxygen as a bridge. Alkali metal cations like sodium (Na⁺) or potassium (K⁺) balance the negative charges on AlO⁴⁻ [1]. The empirical formula of geopolymer may be as below;

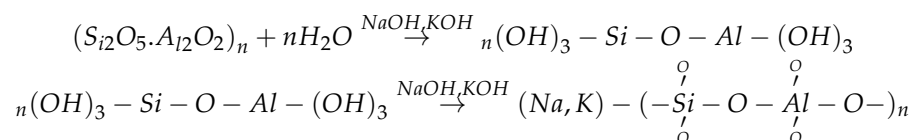


where (M) denotes alkali ions, (n) amount of poly-condensation, and (z) represents silica/alumina in molar ratio [2].

Geopolymer consists of mineral molecular chains bonded to each other with covalent bonding with various molecular units i.e., polysiloxo, polysialate, polysialate-siloxo and polysialate-disiloxo represented as (-Si-O-Si-O-), (-Si-O-Al-O-), (-Si-O-Al-O-Si-O-) and (-Si-O-Al-O-Si-O-Si-O-), respectively [3].

Geo-polymerization is a synthetic reaction involving the reaction of minerals containing alumino-silicate and temperature in a strong basic medium [4], which is a complex process and the mechanism is not completely understood. The mechanism as proposed by Davidovits 1982 may involve the condensation polymerization of ortho sialiate ions

(hypothetical monomers). The silica to alumina ratio may be one or sometimes greater than one. Three steps are involved in polymerization, comprised as follows;



A geopolymer needs three basic components, in which the first one is a reactive alumino silicates source like bottom bagasse ash, meta-kaolin, rice husk ash etc., the second is filler material including thermally activated clay which is also a supplementary source of alumino silicate, and the third one is alkalis such as sodium or potassium hydroxide and their silicates [5]. Bottom bagasse ash is an ideal pozzolana containing ideal properties, which may be used for geopolymer and Portland cement production [6]. High strength geopolymer cement can be synthesized using bagasse ash and clay. Normally, sodium hydroxide and potassium hydroxide or silicates of sodium and potassium are used for geopolymerization. Dissolution of most of the minerals is higher in sodium hydroxide as compared to potassium hydroxide [7]. Moreover, sodium hydroxide demonstrates better resistance than potassium hydroxide towards acids [8]. Furthermore, due to the low cost of sodium hydroxide, geopolymer chemists encourage its use in the synthesis of geopolymers.

Amin et al. synthesized a geopolymer utilizing bottom bagasse ash and studied different parameters [9]. The strength of 50 MPa has been reported using 10 M NaOH with a curing time of 28 days at 80 °C. Kaze et al. [10] explored the kinetic studies in geopolymerization reactions. It has been reported that the concentration of ions like Si⁴⁺ and Al³⁺ affects geopolymer properties. Amin et al. studied the fact that both silicon and aluminum are required for the development of geopolymer with a strong bonded structure. The strength of the geopolymer is influenced by the quantity of alumino-silicate combinations made, which is consecutively affected by the concentration of alkali, temperature, and alumino-silicates available in starting raw material. The relationship of ash fineness with the physico-mechanical properties of high calcium ash-based geopolymer was studied and reported [11]. Geopolymer mortar and concrete has also been synthesized by activating clay having high calcium with a mixture containing 15M sodium silicate and hydroxide [12], and 54.4 MPa strength was achieved at the curing period of 28. The strength was continually increased with the increase in curing time as that of Portland cement mortar and concrete. Zhang et al. [13] studied the curing conditions for the syntheses of the red mud-based geopolymer and found that the strength of the geopolymer rises with an increase in the ratio of both Si/Al and Na/Al. Additionally, a lengthy curing period of up to 180 days was studied which showed a continuous strength increase. Various parameters for the preparation of slag-based geopolymer, including alkali/slag, water/geopolymer, alkaline/water were also studied for the development of maximum compressive strength [13]. He concluded that for excellent strength, concentration of alkali, composition of sodium silicate, ratio of silicate to hydroxide, temperature and curing time are the essential factors. The influence of alkalis, concentration of alkali and activated clay on compressive strength was studied and reported by Cheng et al. [14]. Greater unconstrained compressive strength in short-range curing time was achieved with NaOH and Na₂SiO₃ in the high curing period.

The present work is the synthesis and characterization of a geopolymer synthesized utilizing bottom bagasse ash and thermally activated clay, with ambient environments. The conditions optimized are concentration of alkali, content of alkali and activated clay and curing period. The mechanism of dissolution, strength development of geopolymer, and microstructural and mineralogical studies are studied and reported.

Both of the raw materials such as bagasse ash and clay are abundantly available with no price and their conversion to geopolymer in mixed form is a novel work. Moreover, changing and optimizing the synthetic parameters can result in the production of good

quality geopolymer. The resulting geopolymer is a good alternative for the Portland cement in many applications such as construction material etc.

2. Materials and Experiments

2.1. Raw Materials Characterization

Clay was collected from Nowshera District, khyberpakhtunkhwa, Pakistan. Samples were first dried in the sun, and homogenized by mixing thoroughly. The samples were then dried in the oven at a temperature of 105 °C for a period of 24 h and then stored separately in air tight containers for further study. The physical parameters of the starting raw materials are given in Table 1. Bottom bagasse ash was collected from premier sugar mill Mardan, Pakistan. The ash was pulverized in a mortar and pestle and stored in polythene bags for further studies. The chemical analysis of bagasse ash as studied using XRF is given in Table 1. Flakes of Pure Sodium hydroxide with as high a purity as 98% was purchased from the Merk chemicals and used as alkali activator. A solution of sodium hydroxide in the desired concentration was prepared 24 h prior to its use in order to ensure proper dissolution. The X-ray diffraction pattern of bagasse ash is given in Figure 1. The peaks indicate that bottom ash is composed of quartz and mullite. FTIR spectra of bottom bagasse ash and clay are shown in Figure 2, which shows the main characteristic bands. A peak at 1200–1300 cm^{-1} reveals the presence of CaO in clay, while the peaks in the range of 910–950 cm^{-1} are attributed to the siliceous content confirming that clay also contains some silicates. In bottom bagasse ash, the band at 1030 cm^{-1} represent the mullite phase while that at 1875 cm^{-1} confirms the presence of some water molecules.

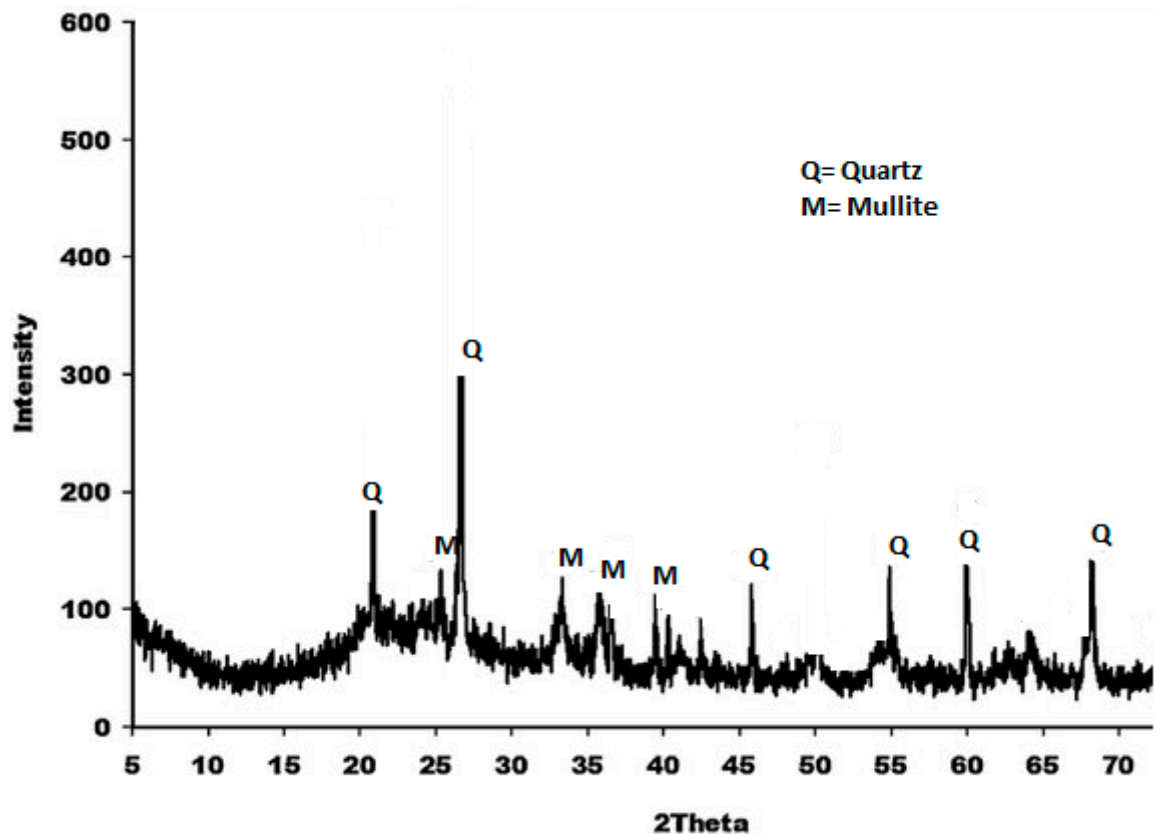
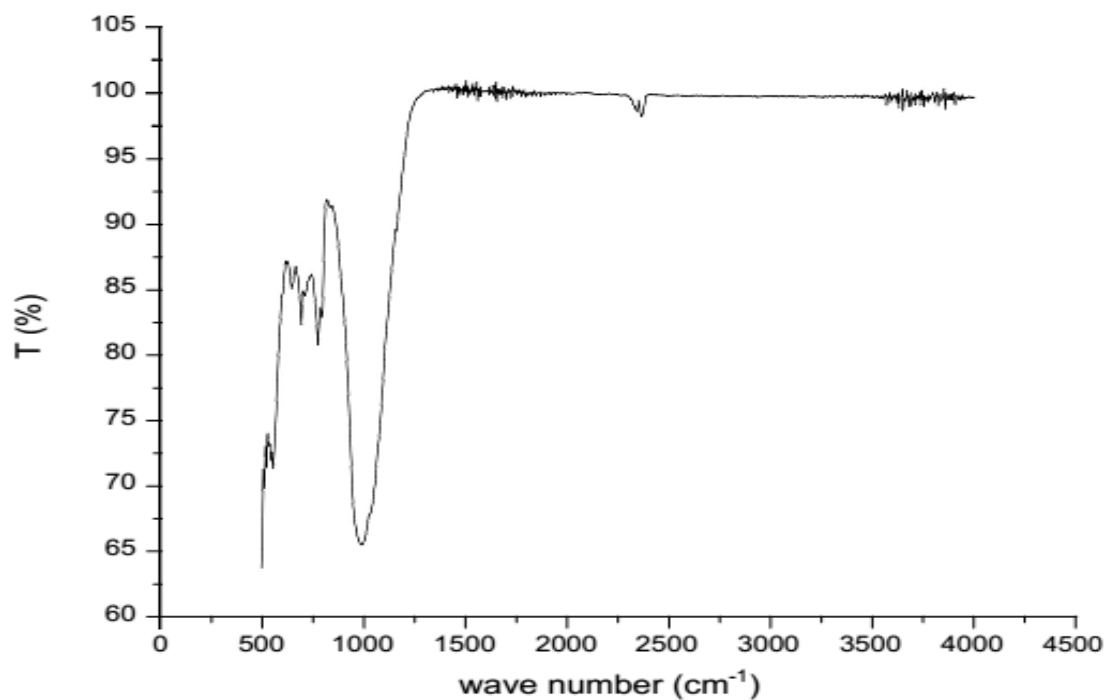


Figure 1. XRD pattern of bagasse ash.

Table 1. Chemical composition of Bagasse ash and China clay.

	wt %							LOI
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	
Bagasse ash	78.046	3.813	2.012	3.975	2.245	0.211	0.421	-
Clay	63.92	17.06	5.88	3.91	1.57	0.14	0.45	1.09

**Figure 2.** FTIR peaks of thermally activated clay.

2.2. Experimental Program

2.2.1. Dissolution of Alumino Silicate from Clay

Dissolved alumino silicate from the clay is different with a different alkaline medium using a Leaching test. For this purpose, 0.5 g of the thermally activated clay was dissolved in 50 mL of NaOH. The concentration of NaOH was kept as 2, 4, 6, 8, 10, 12, 14 and 16 M. The mixture was stirred using a magnetic stirrer at 110 rpm for 20 min. The samples were then filtered using Whatman filter paper 40, and the filtrates were used for Si⁴⁺ and Al³⁺ ions. The amount of Si⁴⁺ and Al³⁺ in the filtrates were studied using Atomic Absorption Spectroscopy (AAS).

2.2.2. Compressive Strength

The clay was mixed with bottom bagasse ash in different ratios in the range of 0, 20, 40, 60, 80 and 100% of activated clay and were named as C₀, C₂₀, C₄₀, C₆₀, C₈₀ and C₁₀₀, respectively. Each blend was mixed with 2, 4, 8 and 16 M NaOH, solution to solid ratio of 0.2, 0.3, 0.4, and 0.5 ml/g. The blends, after thorough mixing, were poured into PVC cylindrical molds and air voids were removed by vibrating the molds for 2 min using a vibrating machine. The specimens were demolded after 24 h of curing, and were kept covered with a plastic sheet to reduce evaporation. Samples of geopolymer were synthesized with various proportions of raw material, various alkali solution concentrations and various ratios of alkali solution to solid. The compressive strength of the whole synthesized geopolymer was studied at 3, 7 and 28 days using the procedure ASTM [15]. Three identical specimens were used for mix proportion, and the average of the three

was considered as the compressive strength. All geopolymer specimens were synthesized, preserved and tested using ambient conditions.

2.2.3. Mineralogical and Structural Analysis

The mineralogical and structural study of the raw materials and synthesized geopolymer was made by an X-ray Diffractometer (XRD) with Cu K α radiation at 40 mA and 40 kV over the range of 5 to 60 at a scanning rate of 5/min. The chemical bonds and structure of the material was studied by Fourier Transform Infra-Red (FTIR) Perkin Elmer Spectrum with a frequency range of 400–4000 cm^{-1} . For this purpose, 13 mm diameter pellets were made by KBr pellet method. A 1 mg sample was ground with 250 mg of KBr and put into pellet form. The pellets were studied at 2 cm^{-1} resolution. For this study, broken pieces of geopolymer after compression strength were kept in ethanol in order to stop further reaction and were used. The ethanol-stored samples were then powdered to less than 75 μm size and then properly homogenized to obtain representative samples.

3. Results and Discussion

3.1. Extent of Si^{4+} and Al^{3+} Ion Dissolution

The dissolution of Si^{4+} and Al^{3+} from clay and bottom bagasse in sodium hydroxide solution with different concentrations is given in Figure 3. The dissolution of Si^{4+} and Al^{3+} occurs from alumino-silicate of the thermally activated clay and bagasse ash, which is drawn-out in the solution. It can be seen in the figure that the leaching of Si^{4+} and Al^{3+} is greater from bottom bagasse ash as compared to that of thermally activated clay, which may be attributed to the fact that bottom bagasse ash comprises a considerable extent of amorphous alumino silicates as compared to thermally activated clay, as is clear from Table 1. Figure 3 also shows that the maximum dissolution of Si^{4+} ions occurs at 8 M NaOH solution, i.e., 160 ppm for clay and 260 ppm for bagasse ash. Above 8M concentration of NaOH, the dissolution again reduces, which is attributed to the rise in agglomeration of silica. Similar phenomena have been observed and reported by some researchers on the dissolution of ions from fly ash samples in alkaline media [11]. Another possible reason for the low dissolution of Si^{4+} at a higher alkali concentration is the low kinetics of ions from the ash and clay, which further arrests the leaching out of ions due to the thickening of the solution. The leaching of Al^{3+} ion at a given alkaline condition is considerably lesser as compared to that of Si^{4+} ion as is clear from Figure 3. The reason for this phenomenon is that both clay and ash have a lower concentration of Al^{3+} ion as compared to that of Si^{4+} ion as is clear from Table 1. Furthermore, Si^{4+} ions have a greater dissolution characteristic than Al^{3+} ions [16]. Like Si^{4+} ion, Al^{3+} ions also show maximum concentration with 8 M NaOH, i.e., 25 and 35 ppm in clay and bottom bagasse ash respectively. The difference in Al^{3+} concentration for bottom bagasse ash, and activated clay is smaller as compared to that of Si^{4+} which depends on the alumina contents in both the ash and clay (Table 1).

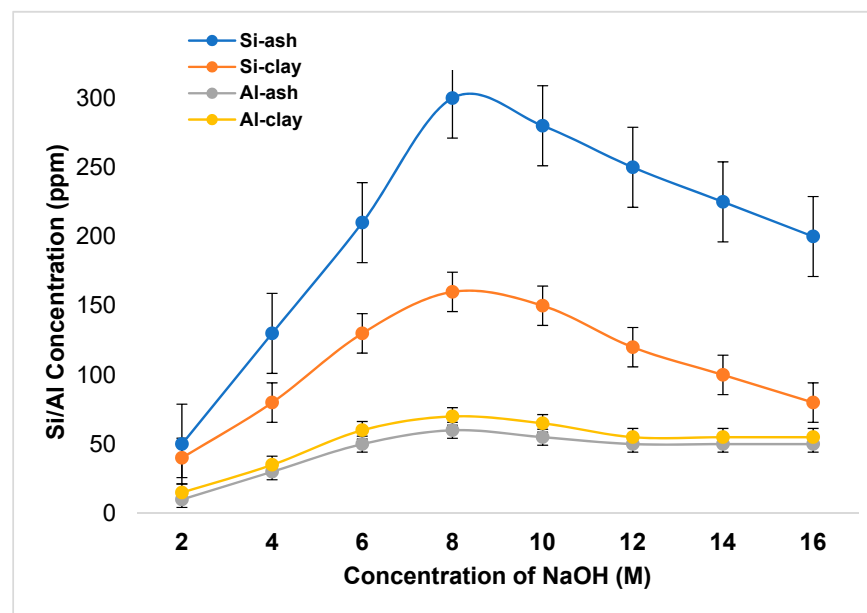


Figure 3. Effect of sodium hydroxide on the dissolution of silica and alumina.

3.2. Effect of Different Parameters on the Compressive Strength

3.2.1. Effect of Alkali Concentration

The concentration of sodium hydroxide has a crucial job in the getting high compressive strength. During our investigations, 28 days of compressive strength was examined with various concentrations of sodium hydroxide, and the results obtained are presented in Figure 4. From the compressive strength results, it is very much clear that the concentration of sodium hydroxide is a significant factor influencing the strength accomplishment. Up to 8 M concentration of sodium hydroxide, the strength expanded essentially, and afterward up to 12 M a decline with further increment is observed. The conceivable clarification for this observation is that OH particles go about as impetus in synthetic response, which quickens the disintegration of alumino-silicate species and Na⁺ particle goes about as charge adjusting cation in the grid [17]. At the point when the grouping of sodium hydroxide is low, lesser Si⁴⁺ and Al³⁺ particles disintegration occurs, which brought about the arrangement of a powerless polymeric system. This results in lower compressive strength. An optimum compressive strength is seen at 8 M NaOH fixation for all the bottom bagasse ash-clay blends. As the leachable Si⁴⁺ and Al³⁺ particles at 8 M NaOH are the greatest, which improves the poly-condensation procedure, it is normal that at 8 M NaOH, the harmony between broken-up types of Si⁴⁺ and Al³⁺ particles and Na⁺ particle is kept up. Subsequently, the most extreme compressive strength is acquired at that concentration. At a focus over 8 M, the draining of alumino-silicates may diminish or remain practically consistent, yet the convergence of free NaOH expands, which brings about a lower silicon to sodium proportion in the network which contrarily influences the poly-condensation procedure, and consequently the strength decreased [18].

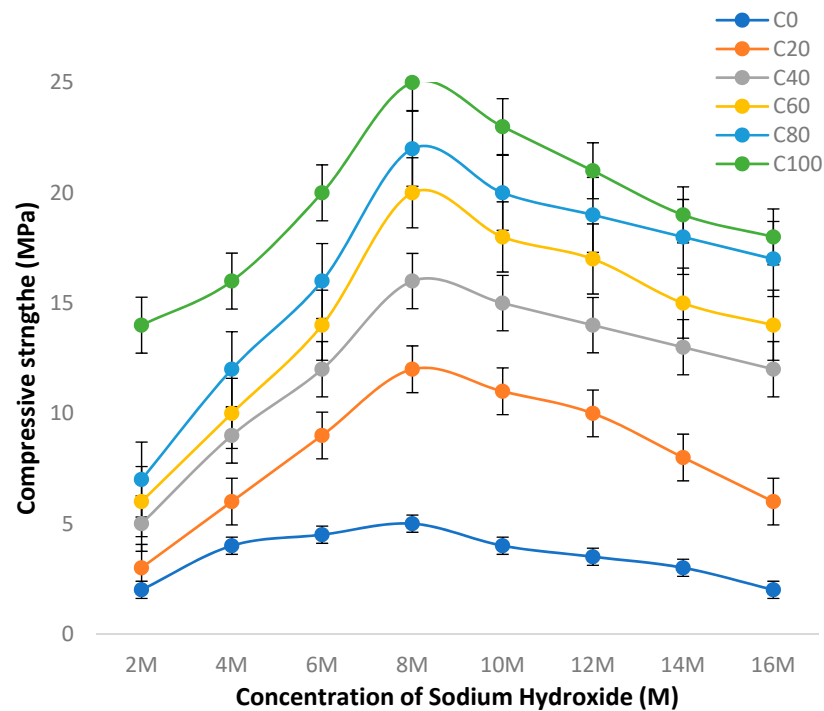


Figure 4. Effect of NaOH on the compressive strength.

3.2.2. Effect of Solution to Solid Ratio

The change of the compressive strength of the geopolymer with a change in ratio of solution to solid is given in Figure 5. For this experiment, the sodium hydroxide concentration used was 8 M, found to be the optimal concentration. It can be perceived from the figure that the strength of the geopolymer is influenced by solution and solid ratio in blend. For all the geopolymer samples at a specific ratio, the strength increases to a certain level and then an adverse effect is observed. The optimum ratio is 0.35 mL/g as it gives maximum compressive strength. A large part of aluminosilicates is crystalline in bottom bagasse ash, whereas it is amorphous in thermally activated clay.

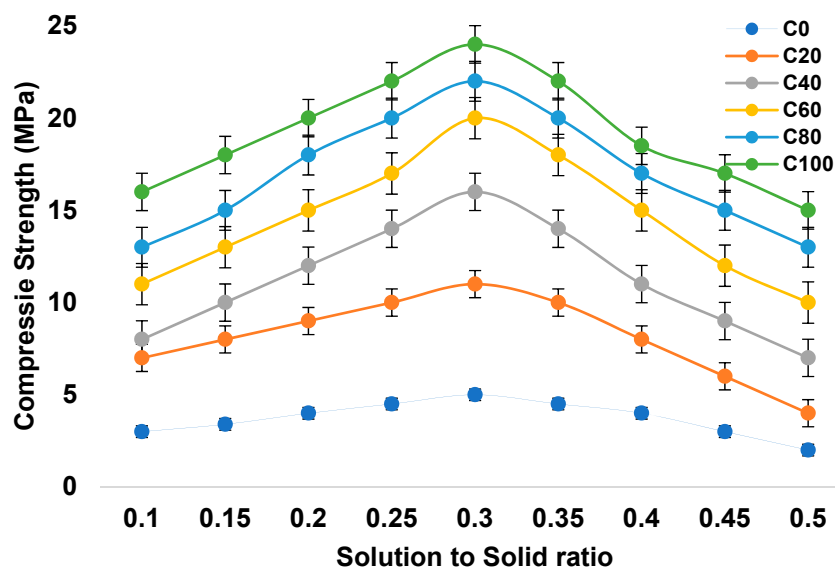


Figure 5. Effect of solution to solid ratio on the compressive strength of geopolymer.

Alkali solution transfers soluble ions like Si^{4+} and Al^{3+} from bottom baggase ash and activated clay into aqueous phase by the dissolution process, which regulates the strength of a geopolymer [13,15], so the amount of alkali used plays a key role in compressive strength improvement of the resulting geopolymer. If the amount of the alkali solution in the matrix is in a smaller quantity, it causes inadequate dissolution of solids, so a poor hydrolysis happens due to a lower solution to solid ratios. In the same way, for a greater ratio of solution to solid, increased dissolution occurs, embedding void spaces after hardening, which obstructs the mechanical veracity and therefore high strength is developed. It should be noted that an excess amount of sodium hydroxide leads to the creation of sodium carbonate as a result of the reaction of extra alkali and carbon dioxide from the atmosphere [19]. Therefore, both the excess/deficiency of alkali solution are not required as it drops the compressive strength. Moreover, excess amounts of Na^+ ions disrupt the balance of charge in the geopolymeric framework. the development of an alumino-silicate polymeric network is the result of poly-condensation, which releases water. There is an additional amount of water leftovers in the craters of structure which is utilized in the hydration reaction in samples of geopolymer samples containing rich activated clay. For samples of geopolymer containing bottom bagasse ash, the polymerization is comparatively slow due to the small discharge of alumino-silicates at normal environmental conditions. Hence, a drop in the compressive strength is detected at a greater ratio of solution/solid, because of the non-availability of an adequate amount of alumino-silicates and improved void spaces in the geopolymeric matrix due to additional molded water.

3.2.3. Effect of Clay Content

The quantity of thermally activated clay in the blend has been found to have extensive impact on the compressive strength of the geopolymer, which has been contemplated following 3, 7, and 28 days curing for all bagasse ash activated clay mixes with 8 M NaOH and solution to solid proportion of 0.35 mL/g and the results are given in Figure 6. A rise in the content of activated clay in blend, causes a rise in the compressive strength. Greater content of the soluble alumino-silicate in alkaline media makes the process of poly-condensation more efficient which helps in the development of a high grade matrix. Therefore, a material with high compressive strength is achieved [19]. Thermally activated clay may comprise typically reactive alumino-silicates and the discharge of ions in alkaline state as compared to that of bottom bagasse ash. It was observed that the strength at 7 and 28 days of curing is increasing with increase in the content of activated clay in blend. Greater content of activated clay in the blend caused more accessibility of ionizable alumino silicates. Moreover, the availability of CaO in the activated clay recruits the hydration process, and consequently the compressive strength of C_{100} is observed to be higher. Moreover, the C_{60} mix showed the highest strength instead of C_{100} for a curing age of 28 days. As for a longer period, the hydration reaction continued in the activated clay, it caused an unceasing dehydration in the geopolymer samples, which resulted in cracks in the specimens due to the shrinkage. Hence, up to a certain proportion of bagasse ash and thermally activated clay blend, enriched compressive strength is observed, and then a decrease in the strength is seen for further increase in the clay content. It is observed that the addition of bottom bagasse ash to activated clay, higher than 30%, reduces the strength of geopolymer samples.

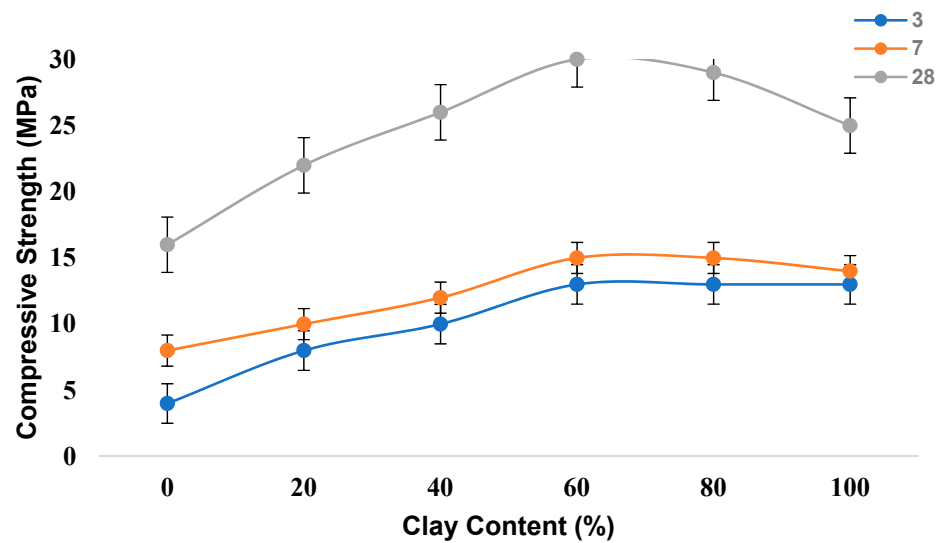


Figure 6. Effect of clay content on the compressive strength of geopolymer.

3.2.4. Effect of Curing Period

Compressive strength development of the geopolymer at various curing ages is shown in Figure 7. During these experiments, the concentration of sodium hydroxide and solution to solid ratio were kept as 8 M and 0.35 mL/g, respectively, as these have been observed as the optimum condition. It is a common observation that curing time is an important parameter for the development of compressive strength. From the figure, it is clear that the relationship between increase in compressive strength and curing period exists almost linear except few samples having thermally activated clay. The compressive strengths of blends C₆₀ and C₁₀₀ become slightly stable after a curing of 7 days. Moreover, after a specific curing time, the samples rich with activated clay show greater compressive strength values as compared to samples having rich bottom bagasse ash. The rate of reaction for bottom bagasse ash is slow as compared to that of activated clay. The existence of minerals with calcium oxide in in clay supports a rapid setting which gives a rapid compressive strength. Similar observations have been reported by many researchers [20].

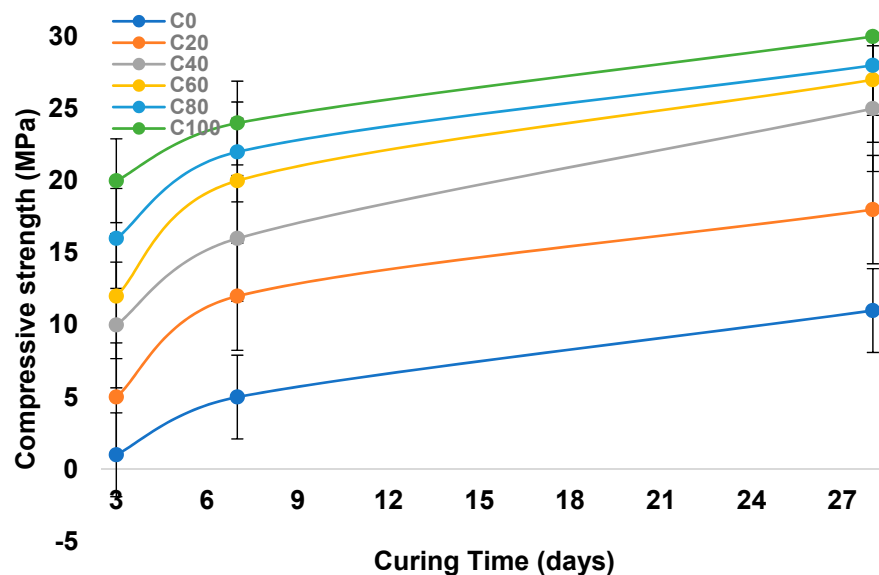


Figure 7. Effect of curing period on the compressive strength of geopolymer.

3.2.5. Confirmation of Chemical Bonds and Reaction Products

The geopolymer product obtained from the reaction of bottom bagasse ash and thermally activated clay, using optimized conditions like 8 M NaOH solution and of 0.35 mL/g solution to solid ratio, cured for 28 days are confirmed from the XRD patterns given in Figure 8. The figure shows that the main products obtained comprise calcium silicate hydrate, calcium aluminium silicate hydrate and calcite.

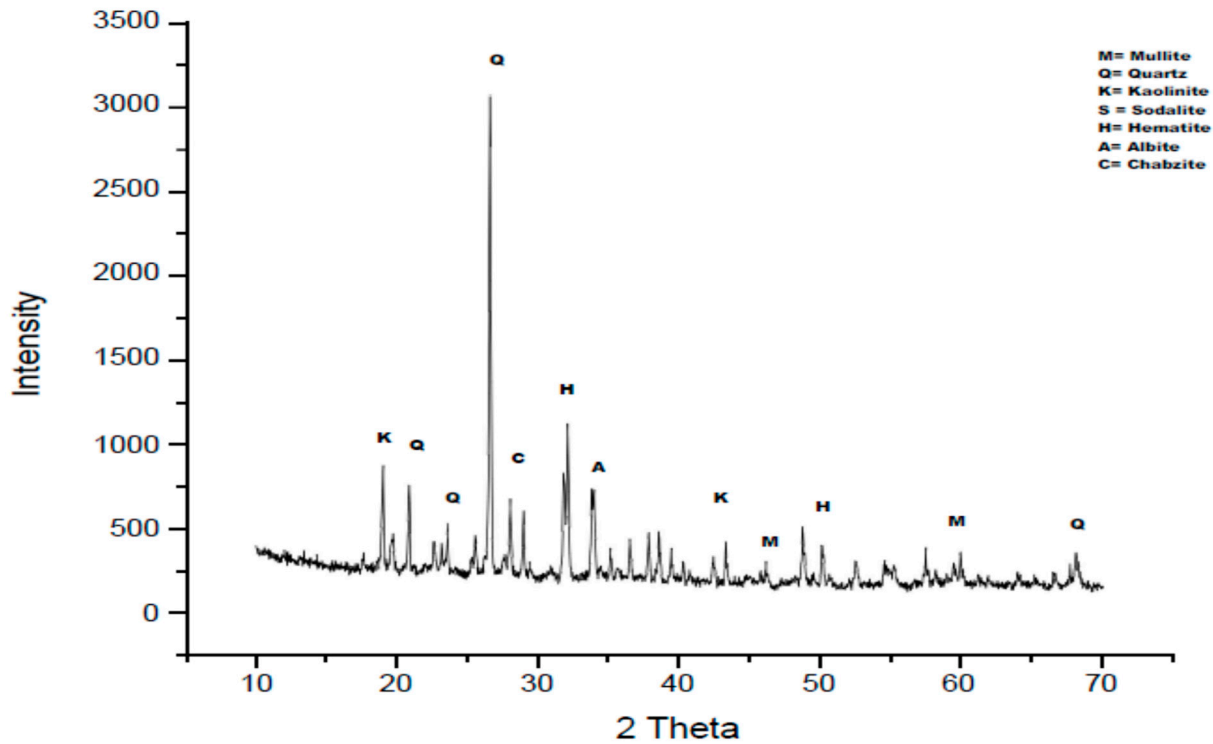


Figure 8. XRD pattern of geopolymer prepared at optimum conditions.

Characteristic peaks of mullite can be seen in bottom bagasse ash (Figure 1) and geopolymer (Figure 8), showing that these peaks remain unchanged during the reaction process. Other small peaks of sodalite, analcime and albite phase can be observed in geopolymer products. Meanwhile, the activated clay-based geopolymer shows large protuberance with extensive and dispersed peaks of semi-crystalline structures.

Figure 9 shows the FTIR spectra of the final product geopolymer prepared under the activated clay-bottom bagasse ash-based geopolymer. The spectra of source material and that of geopolymer products are different from one another by the nature and position of the peaks. The peaks at $685\text{--}898\text{ cm}^{-1}$ correspond to the symmetric stretching-vibration of Si-O-Al while those at 970 and 1087 cm^{-1} are due to the asymmetric stretching of Si-O-Si and Si-O-Al linkages. The silicates and aluminosilicates in the starting material such as bagasse ash and activated clay have contributed to the reaction causing the development of geopolymeric gel which may give high compressive strength, and this phenomenon can be concluded from the shifting of peaks in the raw material. The peaks at $1650\text{--}1800\text{ cm}^{-1}$ of the activated clay-based geopolymer show the existence of some bound water due to the bending vibration of H-O-H. The peaks at $3700\text{--}3760\text{ cm}^{-1}$ may be because of stretching vibrations of OH groups, but the intensity of these peaks is very low in the starting material because there is no reaction product containing calcium- or sodium-based aluminosilicate in the geopolymer.

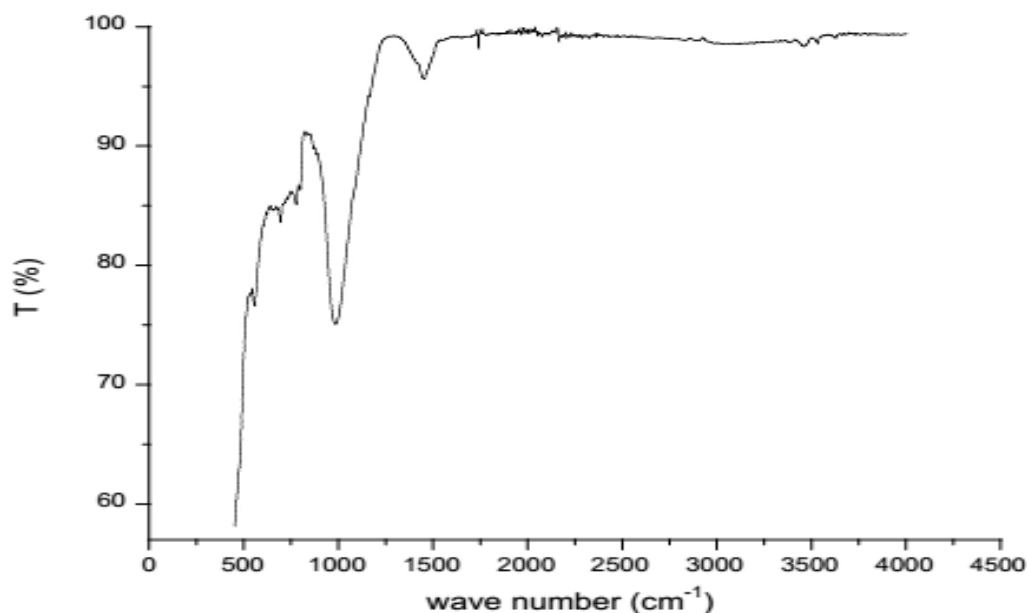


Figure 9. FTIR of the geopolymer obtained at the optimum conditions.

4. Conclusions

The effect of different synthetic parameters on the compressive strength of geopolymer was studied. The leaching mechanism of aluminosilicate ions from both bagasse ash and activated clay with different concentrations of sodium hydroxide was studied. It was found that the leaching of Si^{4+} and Al^{3+} ions from the raw materials depends on the concentration of the NaOH solution and the reactivity of raw material. The increase in compressive strength is observed up to 8M NaOH and then shows a declining trend. Maximum compressive strength is achieved at a solution to solid ratio of 0.3 to 0.35 ml/g. The compressive strength of geopolymer paste increases as the activated clay content in the bagasse ash-clay mix containing 60% activated clay. The reaction products in alkali-activated clay and bottom bagasse ash is different; calcium-based aluminosilicate hydration products are the major binding phase in alkali-activated activated clay with lesser amounts of geopolymeric products. Whereas, the amorphous sodium aluminosilicate is primarily found in alkali-activated bottom bagasse ash with minor amounts of hydration products.

Author Contributions: Basic concept and ideas, N.A.; Methodologies, N.A. and S.S.; Writing, S.A. and A.N.; Figures and Tables, S.G. and N.A.; Supervision, N.A. All authors have read and agreed to the published version of the manuscript.

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