CHAPTER-3

RESULTS AND DISCUSSION

This chapter presents the experimental results conducted in laboratory along with their results and discussions. The experimental tests include Compaction, Strength (Triaxial compressive and Unconfined compressive tests), Hydraulic conductivity, Colorimeter analysis, Durability (Freezing and thawing), and Mineralogical & morphological (XRD and SEM test) on biopolymer stabilized Bauxite residue and Coal mine overburden waste.

3.1 Compaction test

Compaction is used to improve the bearing capacity and stability of the soil. The mixing of biopolymers in this study (individually and in combination) was performed following two methods:

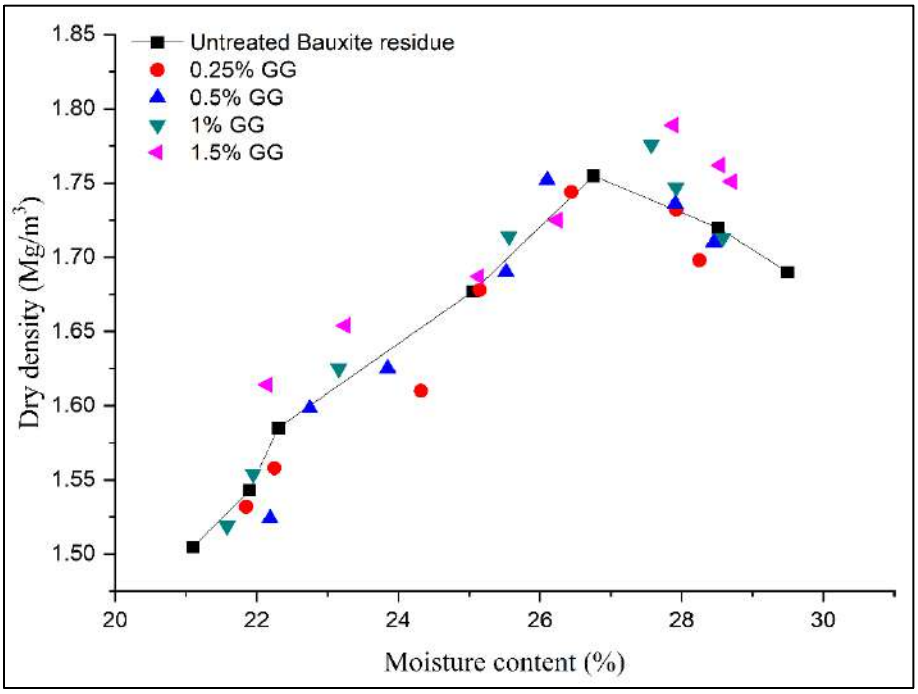
(i) Solution mixing: mixing an already prepared solution of different concentration of biopolymer (i.e., m_b/m_w; biopolymer content to mass of water) with oven-dried sample, and

(ii) Dry mixing: by mixing a biopolymer powder with sample (i.e., m_b/m_{br}; biopolymer content to bauxite residue in mass). In the second method, biopolymer powder was first evenly distributed over the soil layer. Then, water was sprayed on this mixture of soil and biopolymer to reach the desired moisture content. Similar methods of compaction were also followed on silty soil by Karimi (1998).

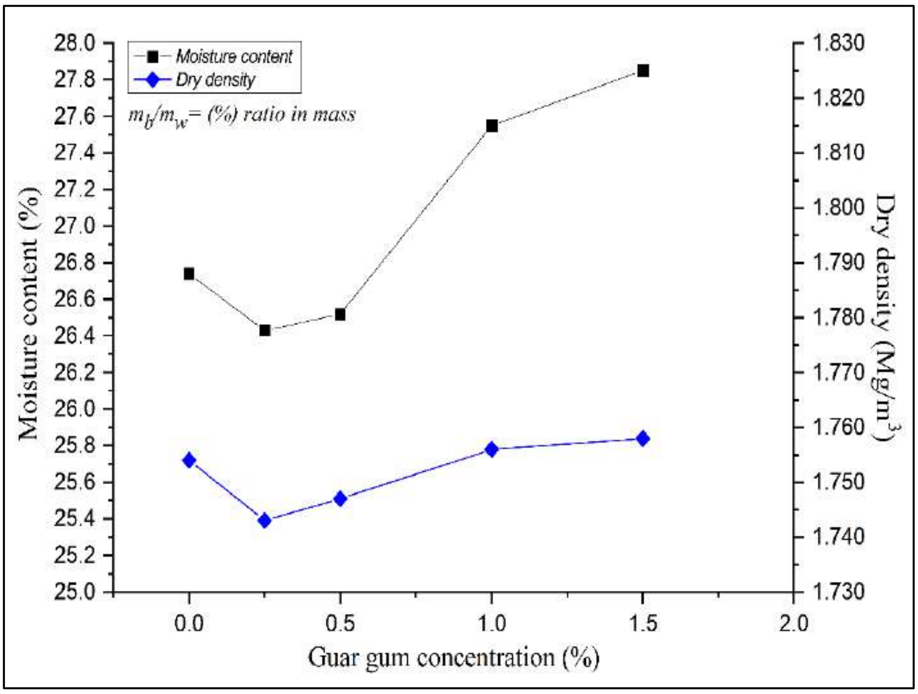
3.1.1 Effect of Biopolymer Stabilization on the Compaction Parameters of bauxite residue

The influence of biopolymer application on compaction characteristics of bauxite residue was evaluated by studying the changes in moisture-density relationship, following the guidelines in ASTM D698. For this, a solution was prepared by mixing xanthan and guar gums, in different concentrations (0.25, 0.5, 1 and 1.5%), individually and as a composite (in equal concentration) with bauxite residue samples and tested. Sample preparation was according to the procedure described in section 2.2. The test results, i.e., the values of maximum dry density (MDD) and the corresponding optimum moisture content (OMC), obtained by two different methods as discussed in section 3.1 are reported in Table 3.1 and shown in Figures 3.1 to 3.6.

The compaction results indicate that the dry density of biopolymer (XG and GG) stabilized bauxite residue sample on the dry side of optimum moisture content is close to untreated bauxite residue sample. However, the dry density tends to increase on the wet side of the optimum. At m_b/m_w =1.5% concentration of XG and GG, the dry density increases from 1.755 (untreated) to 1.822 and 1.789 g/cc respectively; and with composite, it increased to 1.793 g/cc. The higher dry density is due to further reduction in void volume due to the presence of gum particles in the samples, resulting in a dense packing of soil grains. Also, in the dry mixing method, the compaction characteristics are similar to the untreated bauxite residue sample for XG, GG, and composite. This may be possibly due to the mixing of biopolymer in dry form, and water (i.e., at m_b /m_{br} ratio) was added to mixture afterward, the gum particles had not been hydrated enough to generate the bonds between soil particles together. However, in the previous case, the biopolymer solution was hydrated before testing, resulting in samples with higher density.

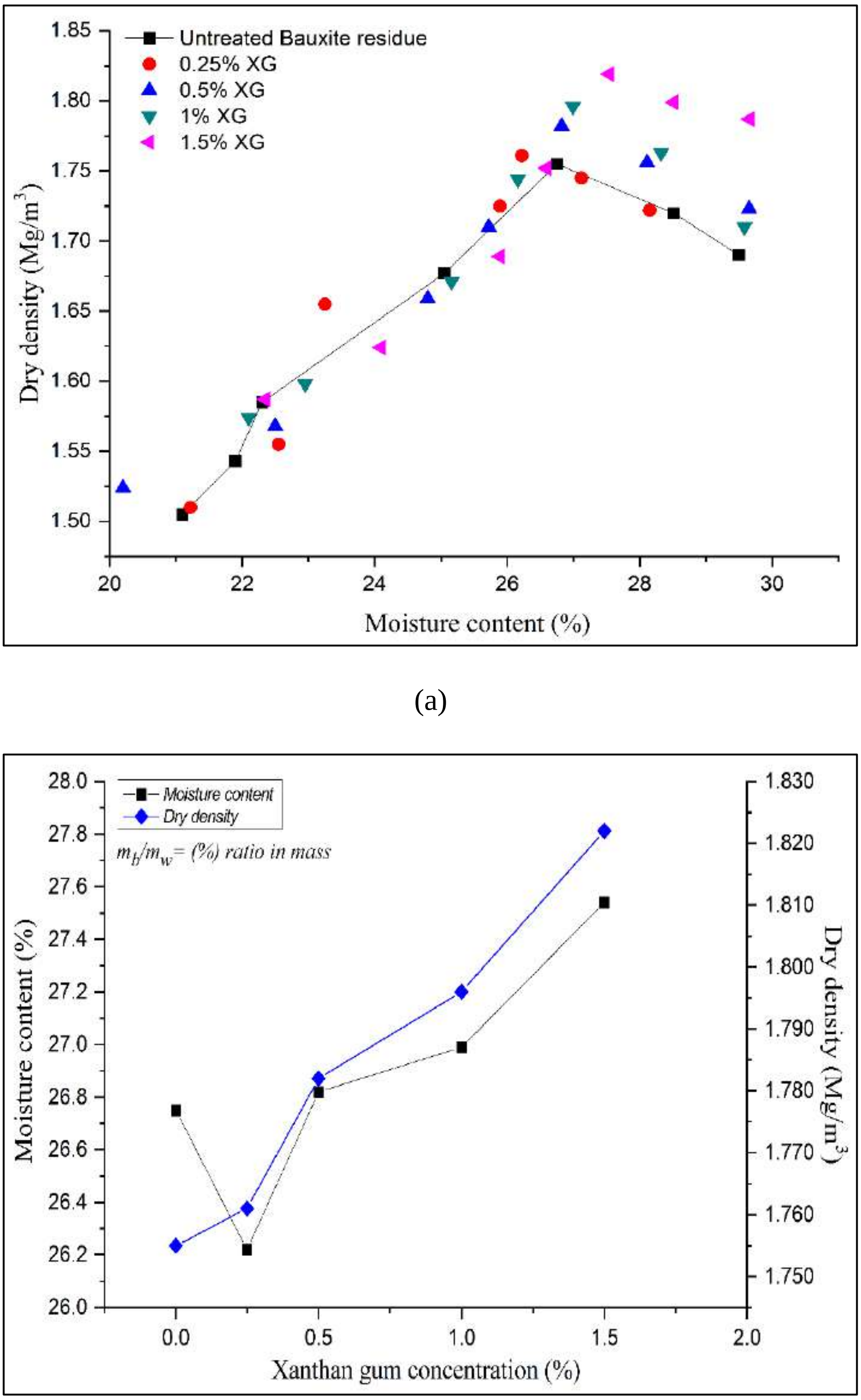


(a)



(b)

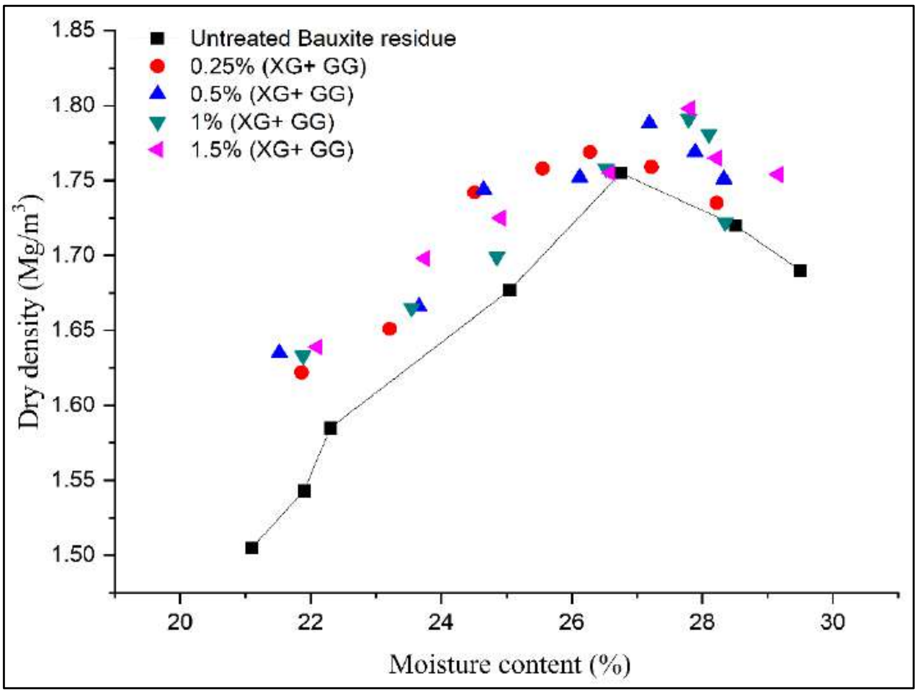
Figure 3.1 Compaction characteristics of bauxite residue stabilized with Guar gum (Method: solution mixing) (a) Effect of guar gum on dry density and moisture content (b) Variation of MDD and OMC with guar gum



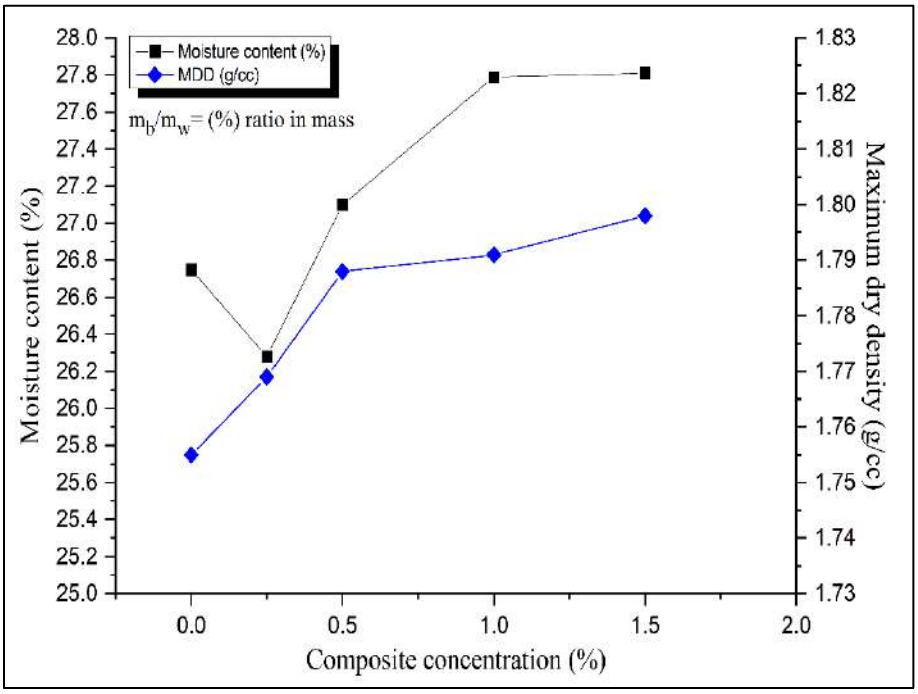
(a)

(b)

Figure 3.2 Compaction characteristics of bauxite residue stabilized with Xanthan gum (Method: solution mixing) (a) Effect of Xanthan gum on dry density and moisture content (b) Variation of MDD and OMC with Xanthan gum

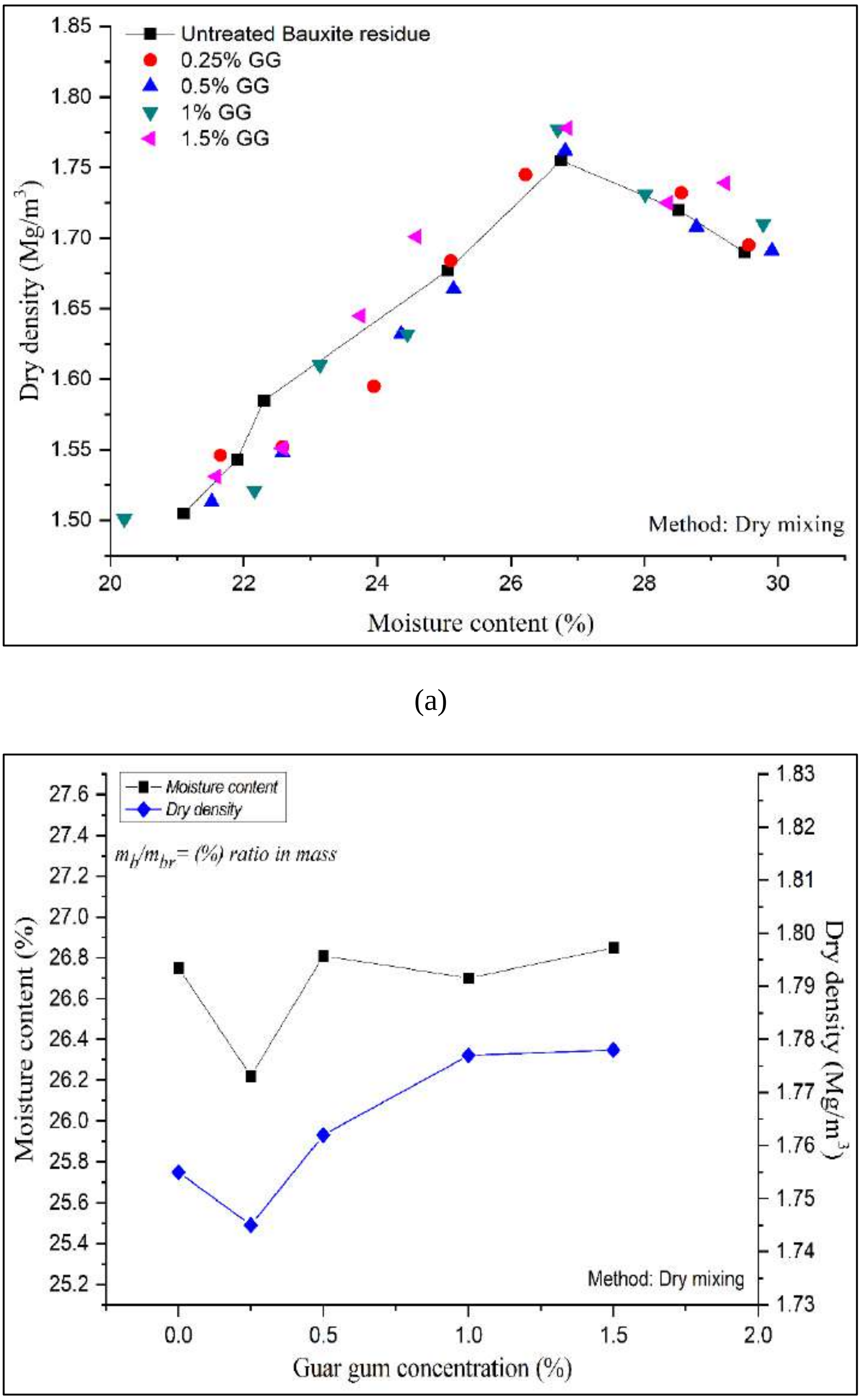


(a)



(b)

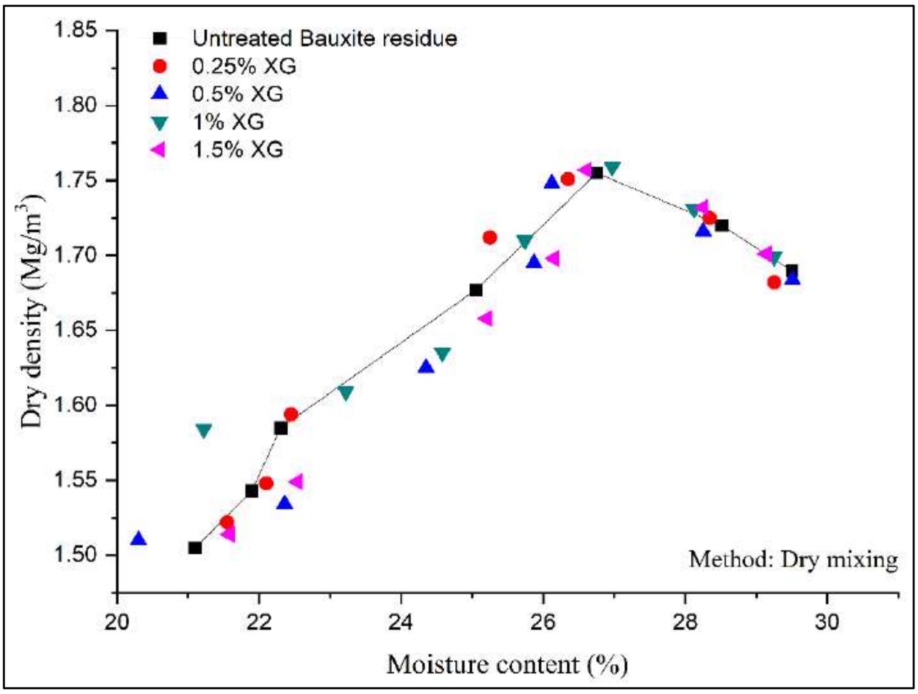
Figure 3.3 Compaction characteristics of bauxite residue stabilized with Composite gum (Method: solution mixing) (a) Effect of Composite gum on dry density and moisture content (b) Variation of MDD and OMC with Composite gum



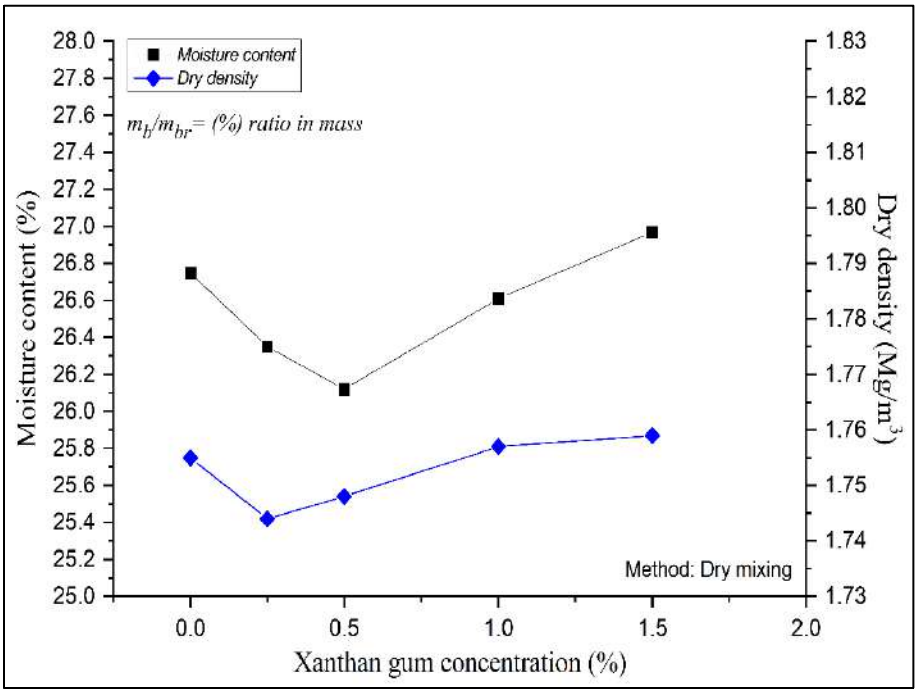
(a)

(b)

Figure 3.4 Compaction characteristics of bauxite residue stabilized with Guar gum (Method: dry mixing) (a) Effect of guar gum on dry density and moisture content (b) Variation of MDD and OMC with guar gum

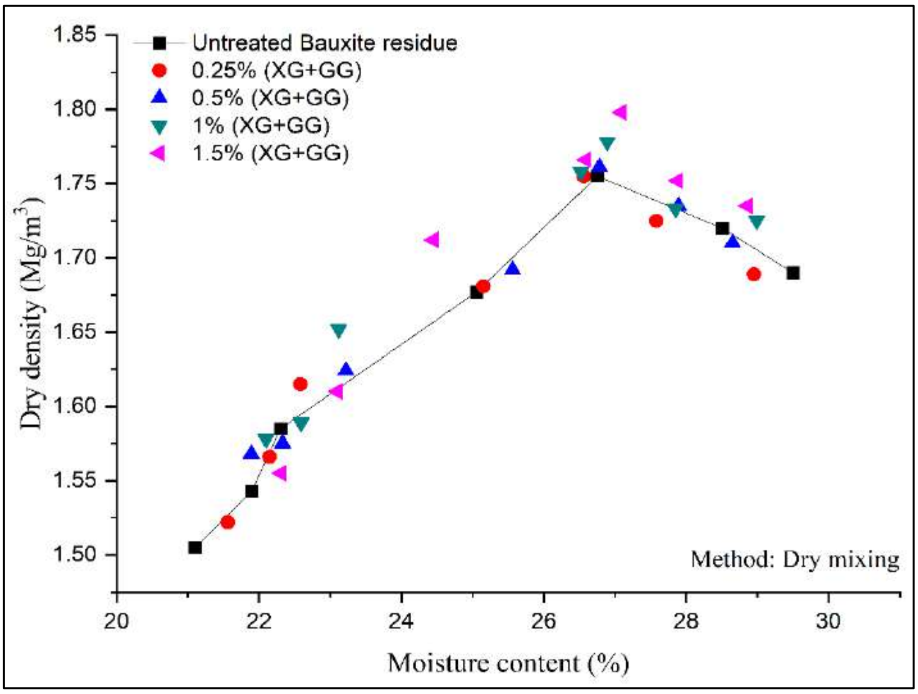


(a)

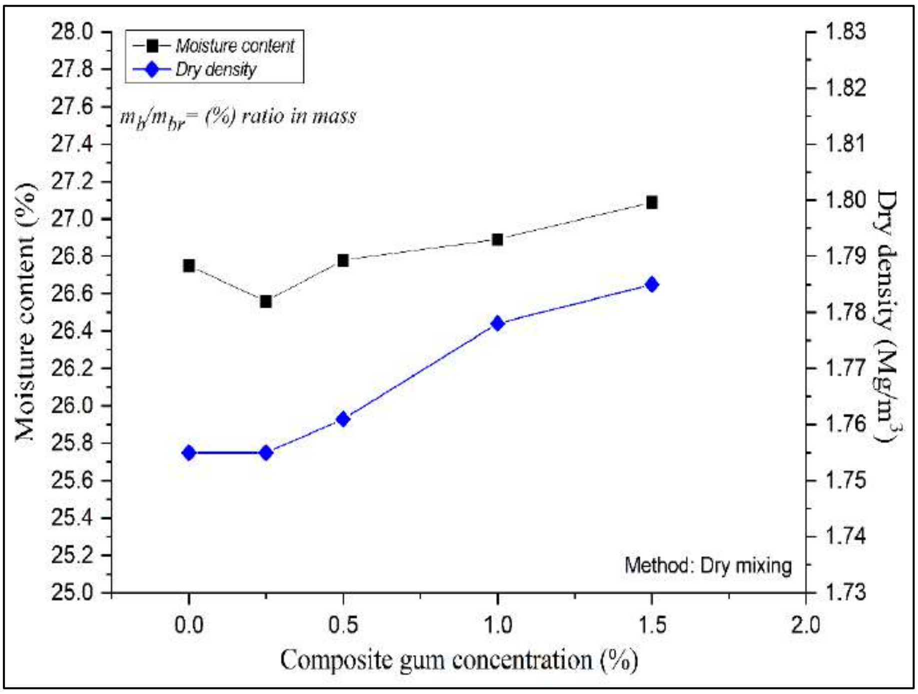


(b)

Figure 3.5 Compaction characteristics of bauxite residue stabilized with xanthan gum (Method: dry mixing) (a) Effect of xanthan gum on dry density and moisture content (b) Variation of MDD and OMC with xanthan gum



(a)



(b)

Figure 3.6 Compaction characteristics of bauxite residue stabilized with Composite gum (Method: solution mixing) (a) Effect of Composite gum on dry density and moisture content (b) Variation of MDD and OMC with Composite gum

Table 3.1 Compaction test results of untreated and stabilized bauxite residue samples

Sample	**OMC (Solution mixing) (%)**	**MDD (Solution mixing) (Mg/m^3)**	**OMC (dry mixing) (%)**	**MDD (dry mixing) (Mg/m^3)**
Untreated bauxite residue	26.75	1.755	26.75	1.755
Bauxite residue + XG (0.25%)	26.22	1.761	26.35	1.754
Bauxite residue + XG (0.5%)	26.82	1.782	26.12	1.748
Bauxite residue + XG (1%)	26.99	1.796	26.61	1.757
Bauxite residue + XG (1.5%)	27.54	1.822	26.97	1.759
Bauxite residue + GG (0.25%)	26.44	1.744	26.22	1.745
Bauxite residue + GG (0.5%)	26.1	1.752	26.81	1.762
Bauxite residue + GG (1%)	27.57	1.776	26.7	1.777
Bauxite residue + GG (1.5%)	27.88	1.789	27.1	1.782
Bauxite residue + XG + GG (0.25%)	26.28	1.769	26.56	1.755
Bauxite residue + XG + GG (0.5%)	27.10	1.788	26.78	1.761
Bauxite residue + XG + GG (1%)	27.79	1.791	26.89	1.778
Bauxite residue + XG + GG (1.5%)	27.81	1.793	27.09	1.785
Bauxite residue + Cement (10%)	17.25	1.761	-	-

3.2 Strength study

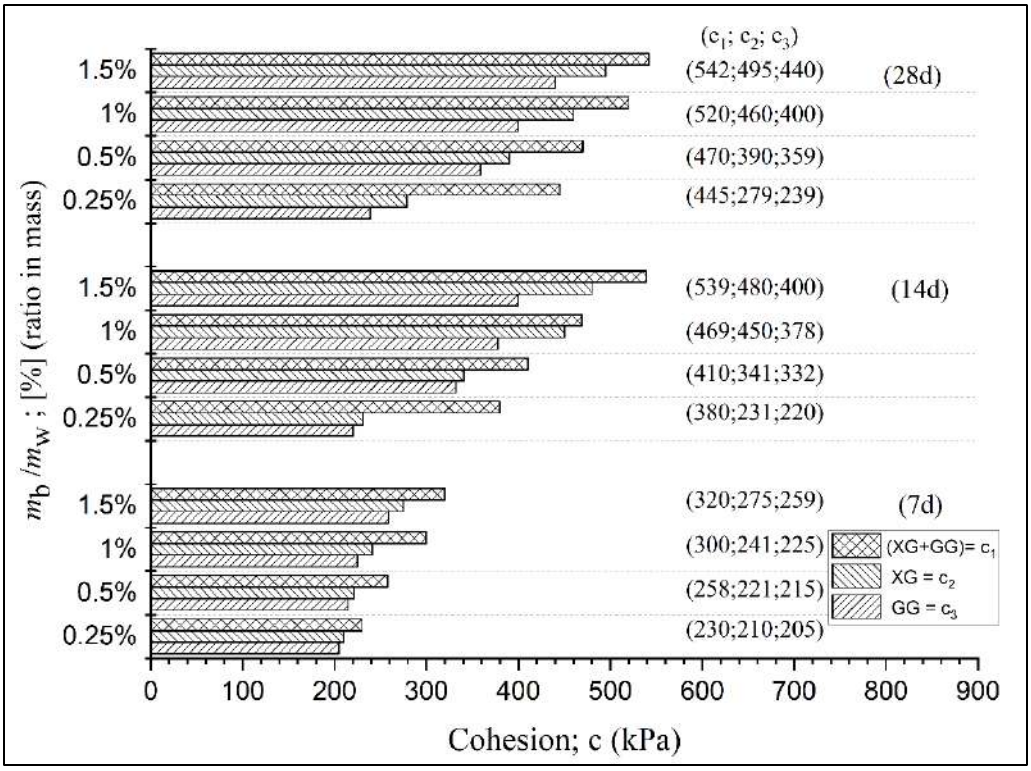
Shear strength and hydraulic conductivity are the essential parameters for reliable and safe design of various geotechnical engineered structures such as slopes, soil barriers, landfill liners, and covers. Enhancing soil/waste characteristics by applying biopolymers capable of binding soil aggregates and therefore increasing the strength and water retention potential is a novel approach that can present an economically and environmentally correct solution. The samples were prepared following the procedure mentioned above (section 3.1) to conduct the triaxial test (UU) and unconfined compression (UCS) test following ASTM D 2850 (2015) and ASTM D2166-06 respectively, as discussed in the section 2.3.6.

3.2.1 Effect of the biopolymer stabilization on Bauxite residue

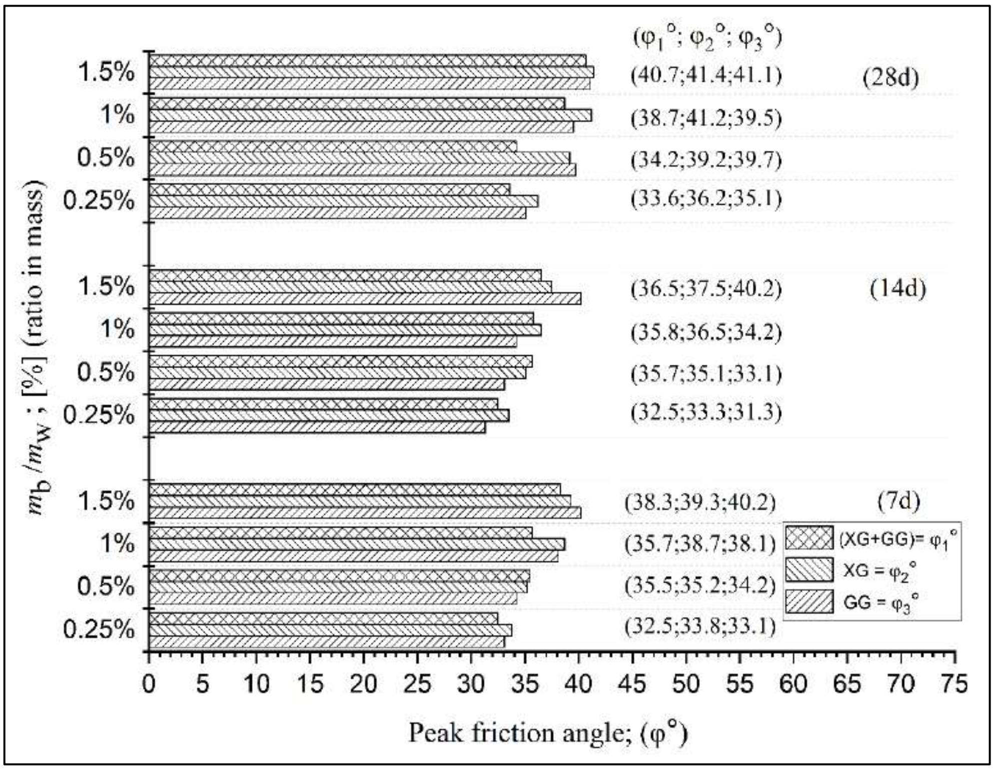
3.2.1.1 Triaxial compressive test

The deviatoric stress-strain responses of biopolymer stabilized bauxite residue at m_b/m_w = 1.5% concentration and 7 d curing period are shown in Figures 4(a) to 4(c). The shear strength parameters of biopolymer stabilized bauxite residue samples evaluated from the triaxial compression test are also shown in Figures 3.7(a) and 3.7(b). The untreated bauxite residue sample (0% biopolymer) exhibited a friction angle, $\varphi = 32^{\circ}$ and cohesion, c = 185 kPa. The biopolymer (individually and as a composite) treatment showed an enhancement in shear strength characteristics with curing time. The failure envelope of the biopolymer amended bauxite residue sample at the varying (m_b /m_w) ratio for 7, 14, and 28 days curing time is shown in Figures 3.8(a) to 3.8(i). A large shift of the failure envelope in the upward direction can be seen for the stabilized bauxite residue sample compared to the untreated one (0% biopolymer), implying an increase in shear strength. It was observed to improve with the increase in concentration (m_b /m_w) from 0.25 to 1.5% with the composite showing higher increment compared to xanthan and guar gum when used individually. At 28 days of curing, 1.5% of biopolymer (GG, XG, and composite (XG+GG)) showed 226, 259, and 275% increase in shear strength of bauxite residue. The biopolymer amended bauxite residue samples showed an increase in cohesion from 185 to 542 kPa and friction angle from 32 to 41°. This increase can be attributed to the interconnections among soil particles developed by biopolymer addition that increased with an increase in its concentration and curing time. It indicates that the curing time also plays a crucial role in enhancing the shear strength characteristics of biopolymer amended bauxite residue samples. The maximum enhancement was observed for 28 days cured samples, for all concentration of biopolymers.

It is also important to note that treatment with a composite biopolymer (Xanthan and Guar gum) showed higher enhancement than biopolymers used individually, possibly due to the formation of an interpenetrating cross-linked network. It is more difficult to break cross-linked gels chemically because the breaking agent must react with the cross-linked bond to the polymer molecule, in addition to attacking the polymer backbone. Similar findings have been reported elsewhere [1]. Also, the nature of hydrogel bonding with soil particles depends on biopolymer type; thus, the enhancement in strength of xanthan gum treated bauxite residue has a greater impact compared to guar gum possibly due to guar gum being a neutrally charged polysaccharide in the absence of carboxylic acid groups, whereas xanthan gum has an electric charge that interacts with cations of the clay portion of the sample resulting in chemically stronger ionic bonds in addition to hydrogen bonds, leading to increased resistance [78].

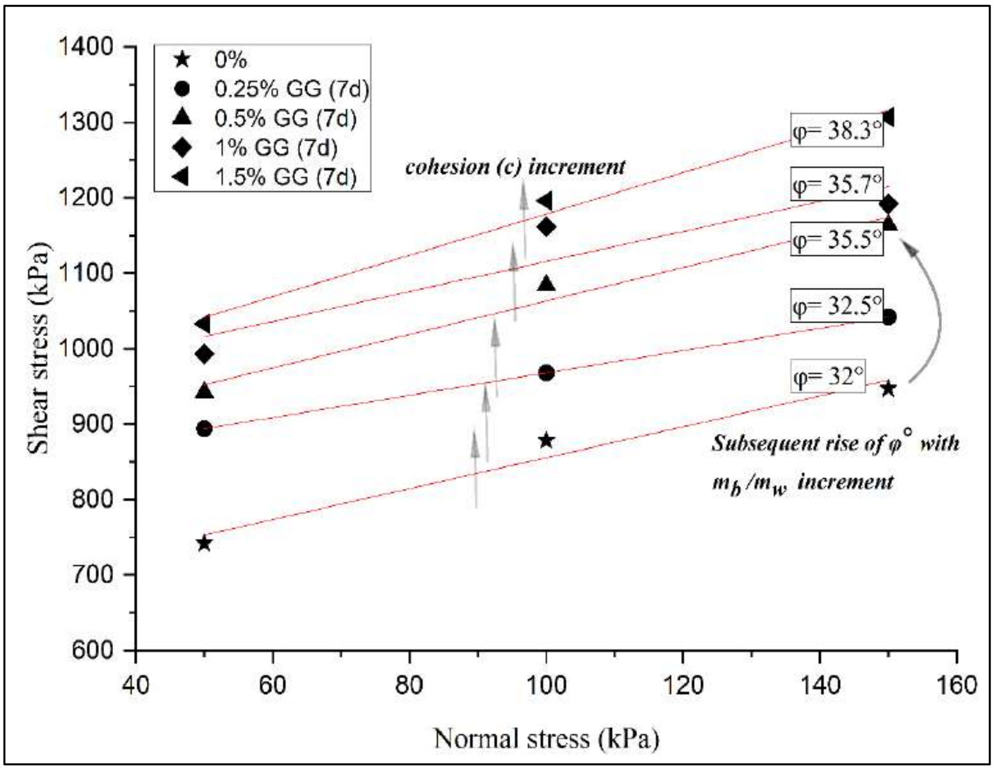


(a)

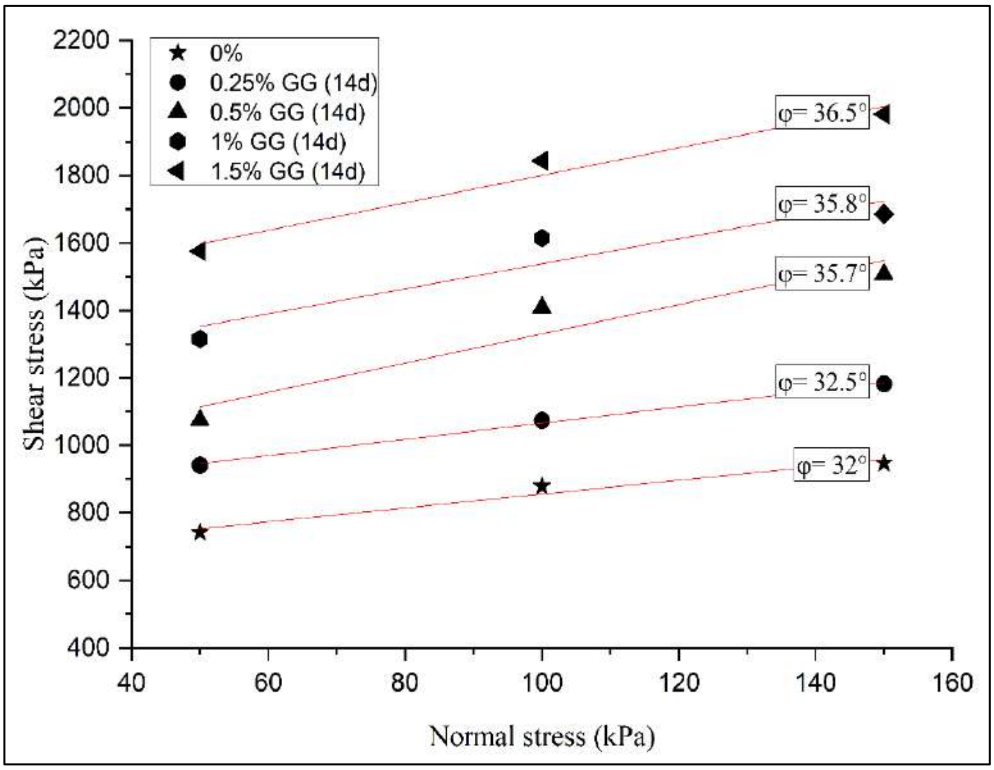


(b)

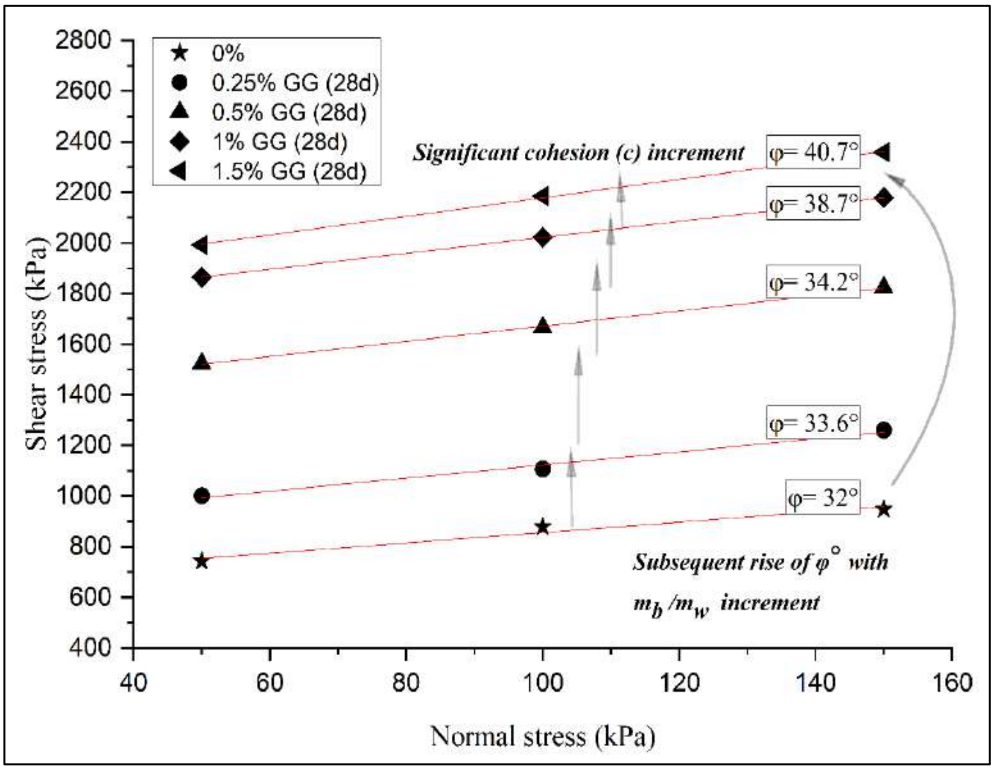
Figure 3.7 Shear strength parameters of Bauxite residue treated with different concentration (m_b/m_w) [%] of biopolymer solution (GG, XG and composite) at different curing time (28d; 14d; 7d): (a) Cohesion (c), (b) Peak Friction angle (ϕ°)


★ 0%
● 0.25% GG (7d)
▲ 0.5% GG (7d)
◆ 1% GG (7d)
◀ 1.5% GG (7d)
cohesion (c) increment
φ= 38.3°
φ= 35.7°
φ= 35.5°
φ= 32.5°
φ= 32°
Subsequent rise of φ° with
m_b/m_w increment
Shear stress (kPa)
1400
1300
1200
1100
1000
900
800
700
600
40
60
80
100
120
140
160
Normal stress (kPa)

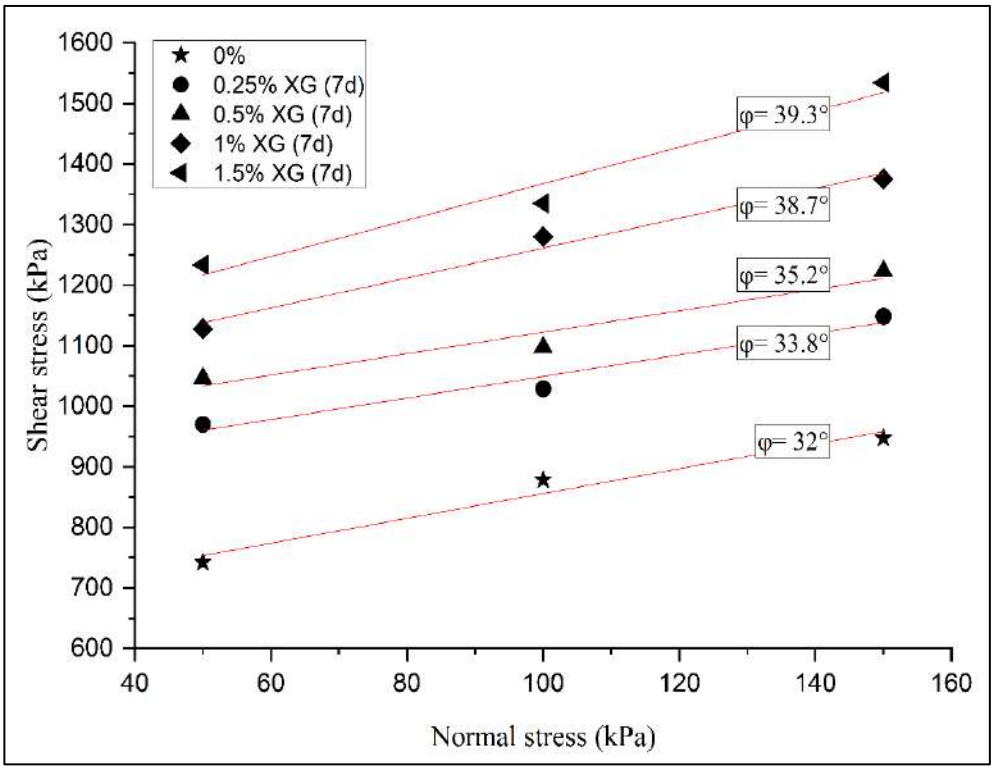
(a)


★ 0%
● 0.25% GG (14d)
▲ 0.5% GG (14d)
⬢ 1% GG (14d)
◀ 1.5% GG (14d)
φ= 36.5°
φ= 35.8°
φ= 35.7°
φ= 32.5°
φ= 32°
Shear stress (kPa)
2200
2000
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160
Normal stress (kPa)

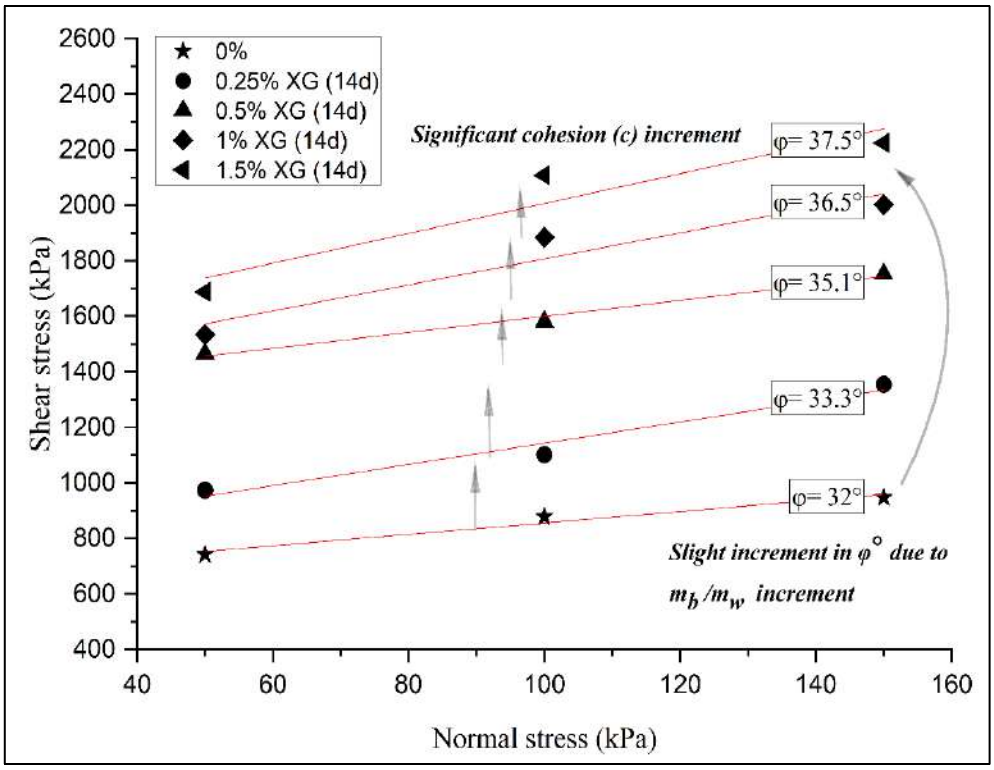
(b)


Shear stress (kPa)
Normal stress (kPa)
0%
0.25% GG (28d)
0.5% GG (28d)
1% GG (28d)
1.5% GG (28d)
Significant cohesion (c) increment
φ= 40.7°
φ= 38.7°
φ= 34.2°
φ= 33.6°
φ= 32°
Subsequent rise of φ° with
m_b /m_w increment
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160

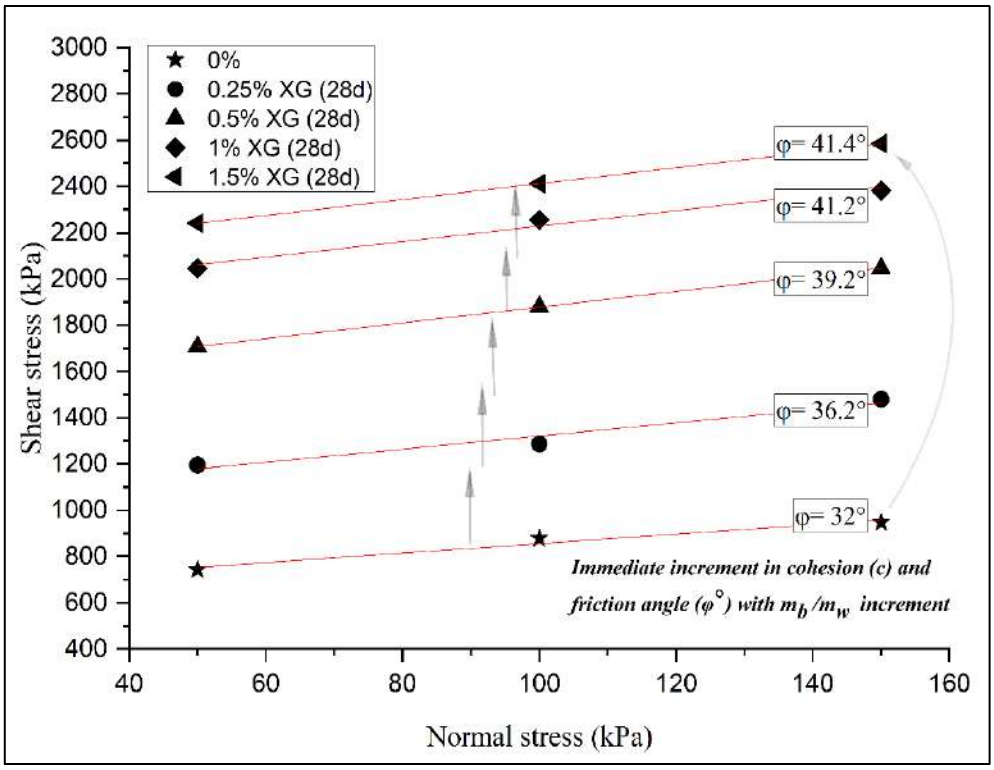
(c)


Shear stress (kPa)
Normal stress (kPa)
0%
0.25% XG (7d)
0.5% XG (7d)
1% XG (7d)
1.5% XG (7d)
φ= 39.3°
φ= 38.7°
φ= 35.2°
φ= 33.8°
φ= 32°
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
40
60
80
100
120
140
160

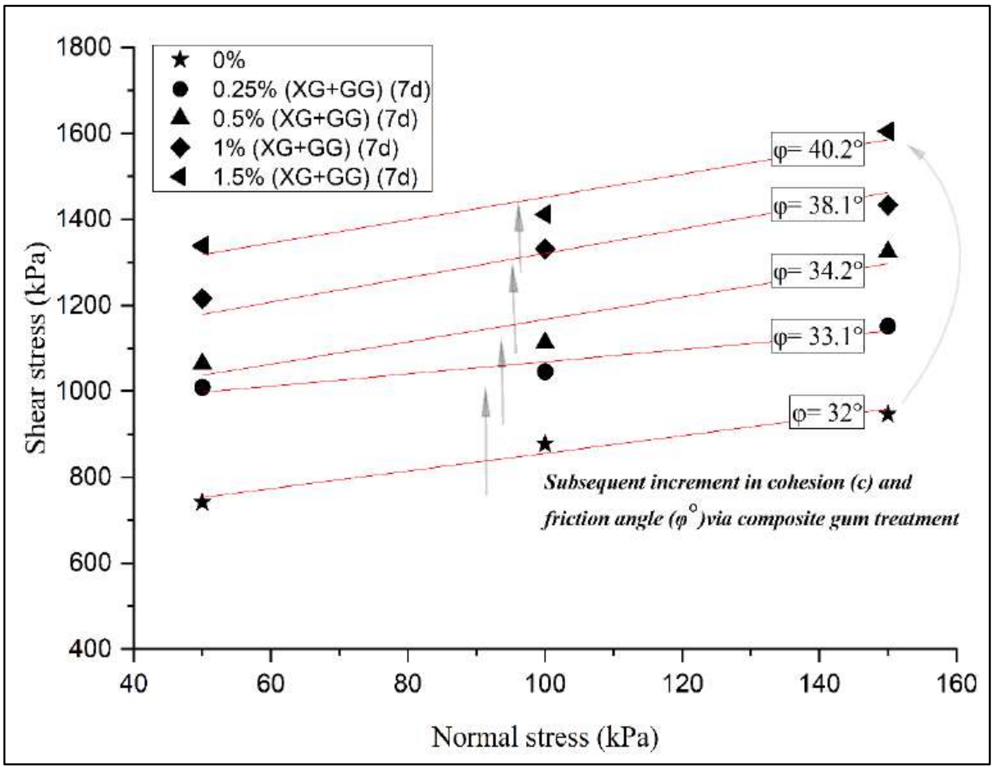
(d)


★ 0%
● 0.25% XG (14d)
▲ 0.5% XG (14d)
◆ 1% XG (14d)
◀ 1.5% XG (14d)
Significant cohesion (c) increment
φ= 37.5°
φ= 36.5°
φ= 35.1°
φ= 33.3°
φ= 32°
Slight increment in φ° due to
m_b/m_w increment
Shear stress (kPa)
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160
Normal stress (kPa)

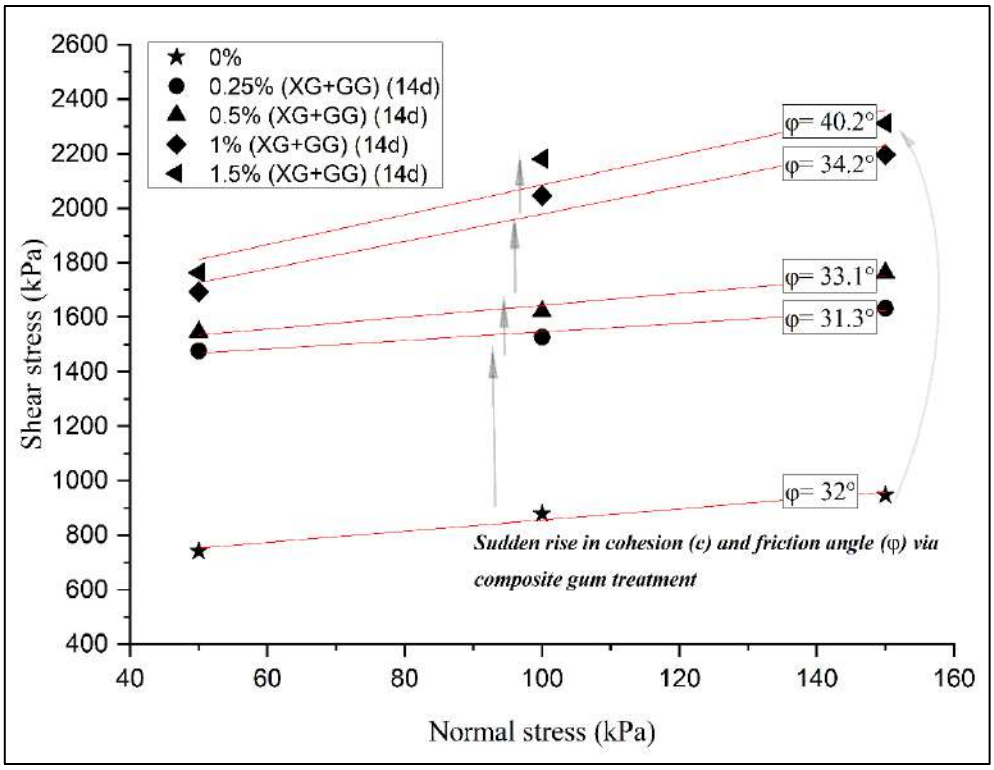
(e)


★ 0%
● 0.25% XG (28d)
▲ 0.5% XG (28d)
◆ 1% XG (28d)
◀ 1.5% XG (28d)
φ= 41.4°
φ= 41.2°
φ= 39.2°
φ= 36.2°
φ= 32°
Immediate increment in cohesion (c) and
friction angle (φ°) with m_b/m_w increment
Shear stress (kPa)
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160
Normal stress (kPa)

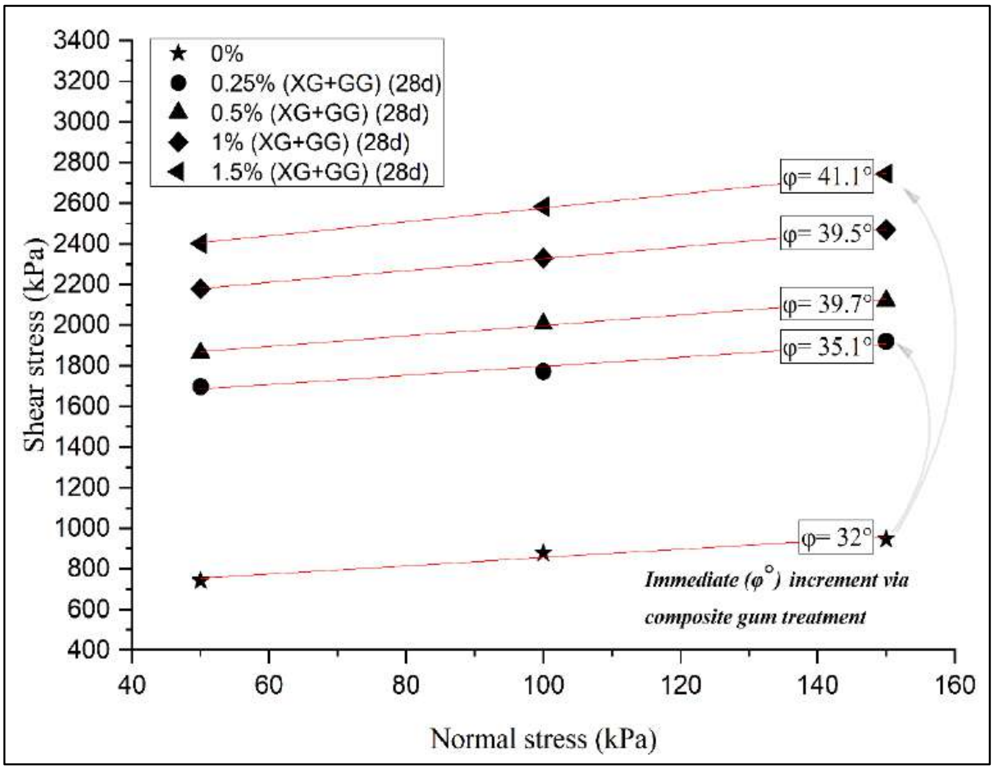
(f)


0%
0.25% (XG+GG) (7d)
0.5% (XG+GG) (7d)
1% (XG+GG) (7d)
1.5% (XG+GG) (7d)
φ= 40.2°
φ= 38.1°
φ= 34.2°
φ= 33.1°
φ= 32°
Subsequent increment in cohesion (c) and friction angle (φ°)via composite gum treatment
Shear stress (kPa)
Normal stress (kPa)
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160

(g)


0%
0.25% (XG+GG) (14d)
0.5% (XG+GG) (14d)
1% (XG+GG) (14d)
1.5% (XG+GG) (14d)
φ= 40.2°
φ= 34.2°
φ= 33.1°
φ= 31.3°
φ= 32°
Sudden rise in cohesion (c) and friction angle (φ) via composite gum treatment
Shear stress (kPa)
Normal stress (kPa)
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
40
60
80
100
120
140
160

(h)

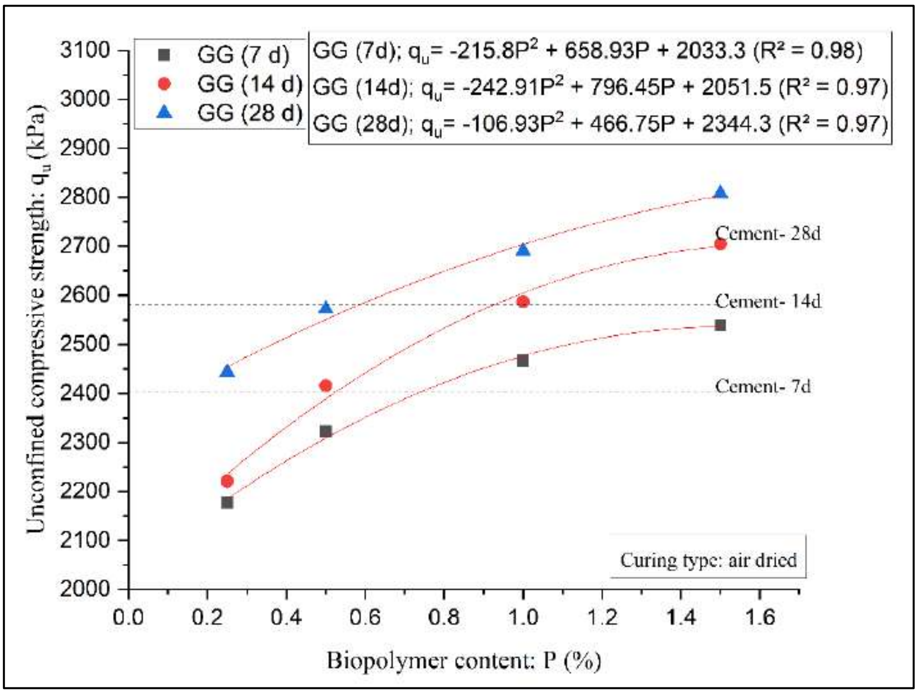


(i)

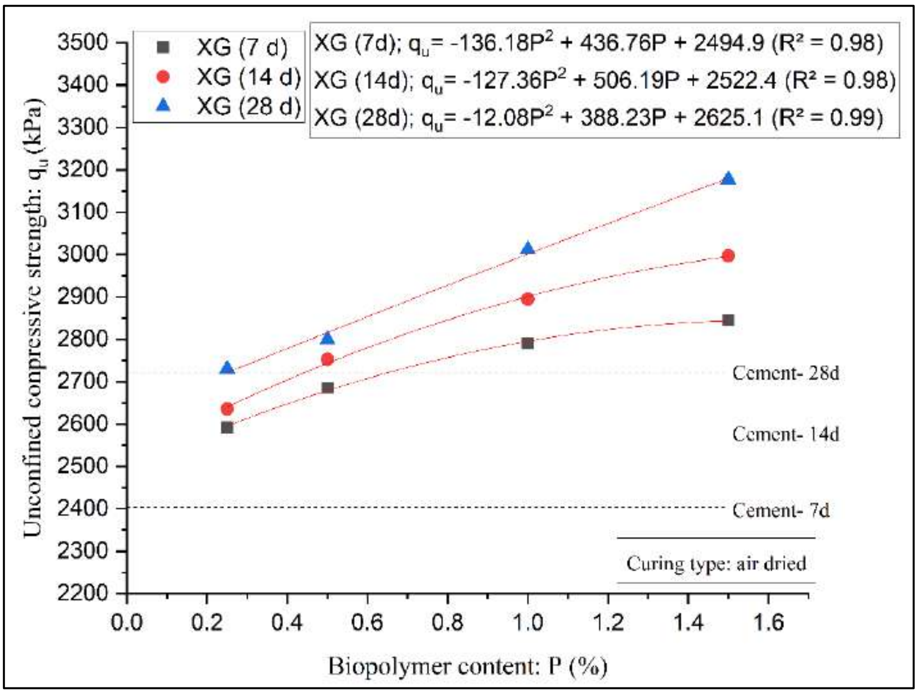
Figures 3.8 Failure Envelope of Stabilized Bauxite Residue Sample for Varying Biopolymer Concentration (m_b/m_w) at Different Curing Time: (a) GG (7d), (b) GG (14d), (c) GG (28d), (d) XG (7d), (e) XG (14d), (f) XG (28d), (g) XG+GG (7d), (h) XG+GG (14d), (i) XG+GG (28d)

3.2.1.2 Effect of biopolymer, biopolymer concentration and curing time on the UCS and stress-strain behavior

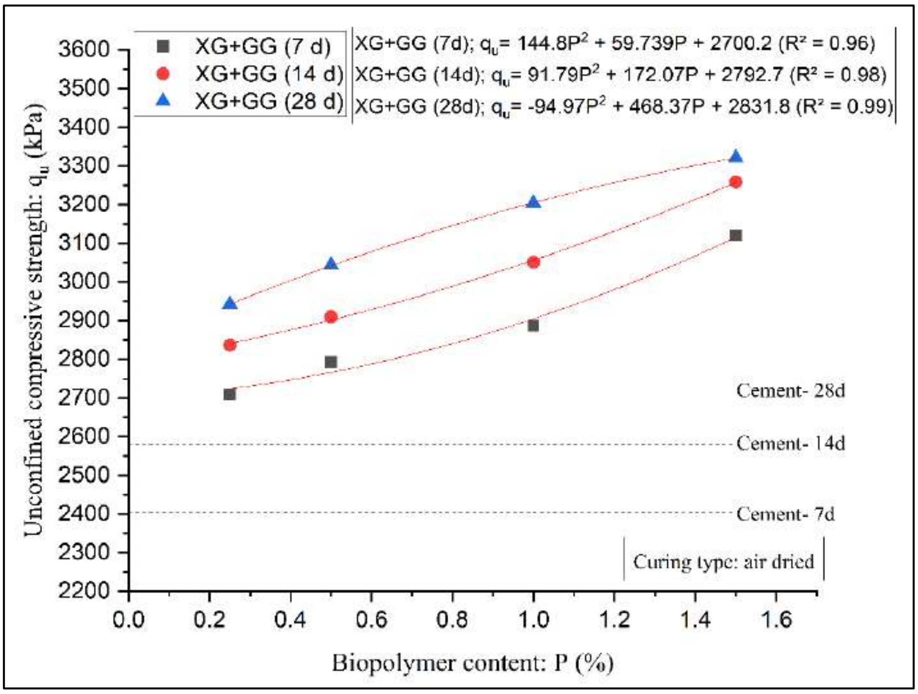
The unconfined compressive strength (UCS) of the biopolymer stabilized bauxite residue are shown in Figure 3.9. The UCS test response of 10% cement-treated bauxite residue specimen is also plotted therein. After 7, 14 and 28 days of curing, there is a considerable increase in the UCS at higher biopolymer concentration. The UCS of untreated bauxite residue is 247 kPa, and the addition of GG, XG, and composite at m_b/m_w = 1.5% concentration induces considerable higher strength of 2808, 3177, and 3321 kPa respectively. It can be seen that the addition of 1.5% composite biopolymer provides bauxite residue samples with compressive strength higher than the 10% cement-treated sample at 29.8% higher after seven days, 26.2% higher after 14 days and, 22% higher after 28 days. Also, the peak strength (average) of bauxite residue amended with 0.5% XG at 28 days (2,800 kPa) is comparatively higher than 10% cement-treated sample after the same curing time (2,720 kPa). It depicts that either 100 kg of cement (10% of the bauxite residue) or 5 kg of xanthan gum (0.5% of the bauxite residue) would be required to enhance the strength of bauxite residue over 2.7 MPa. Similar behavior in the enhancement of strength of gellan gum treated sand was also reported by Chang et al. (2016).



(a)



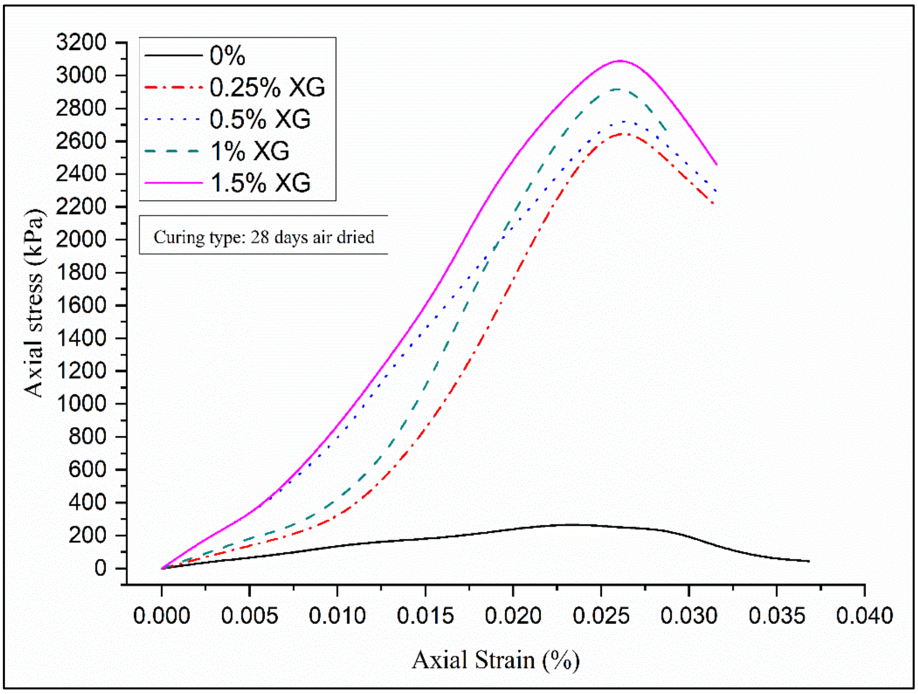
(b)



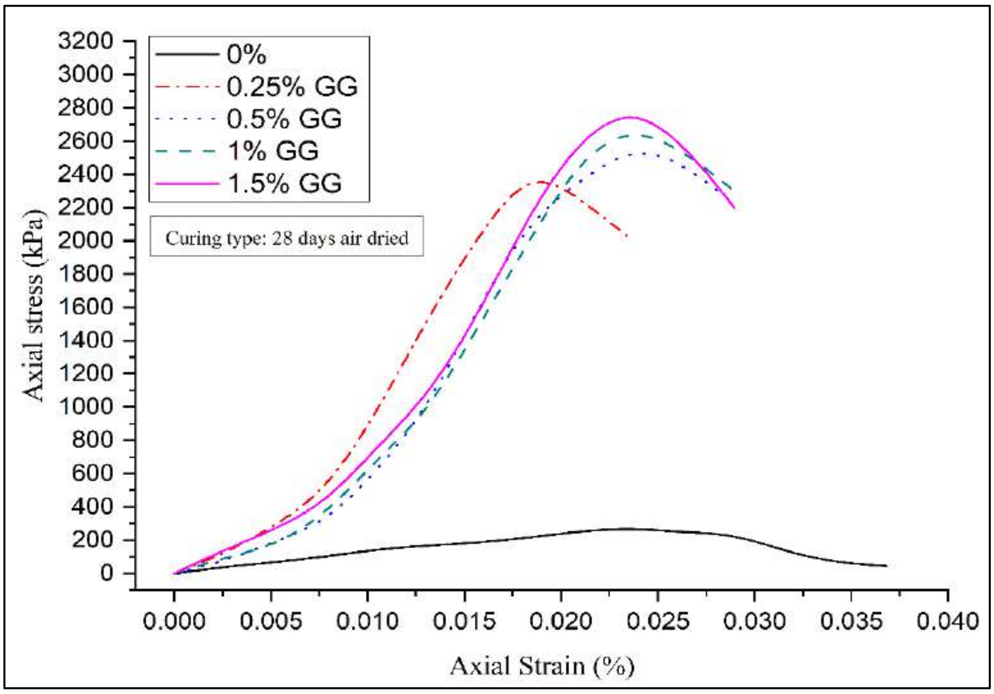
(c)

Figure 3.9 Variation in UCS (q_u) with Biopolymer, Biopolymer Content (P %) and Curing Time (d): (a) Guar Gum, (b) Xanthan Gum, (c) Composite (XG+GG)

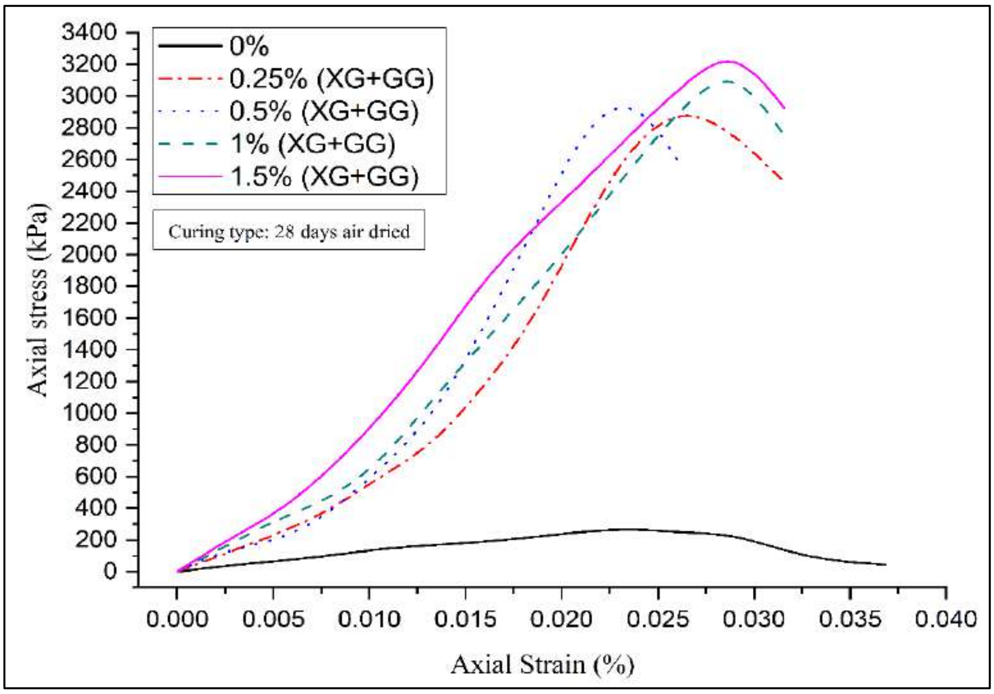
The improved strength of the bauxite residue sample is due to dehydration of residual moisture in biopolymer gel resulting in micro-interactions of particles that enhanced the inter-particle contact characteristics. Their retention is due to the adsorption mechanism of polymer emulsion that causes higher cohesion within the bauxite residue particles, increasing the UCS values. Therefore, the use of a biopolymer for soil/waste treatment can also be highly recommended under low moisture conditions. The stress-strain plots obtained from the UCS test for various concentrations of XG, GG and composite and curing periods are shown in Figures 3.10 (a) to (c), respectively. It is observed that the residual response of the specimen treated with higher biopolymer content is more significant compared to samples treated with smaller content. Similarly, with higher curing periods, the sample's stiffness also increases (Figure 3.11). It means that with an increase in biopolymer concentration and curing time, the bauxite residue samples show greater strength at the same strain level. Also, a similar response was observed for the GG and composite (XG + GG).



(a)

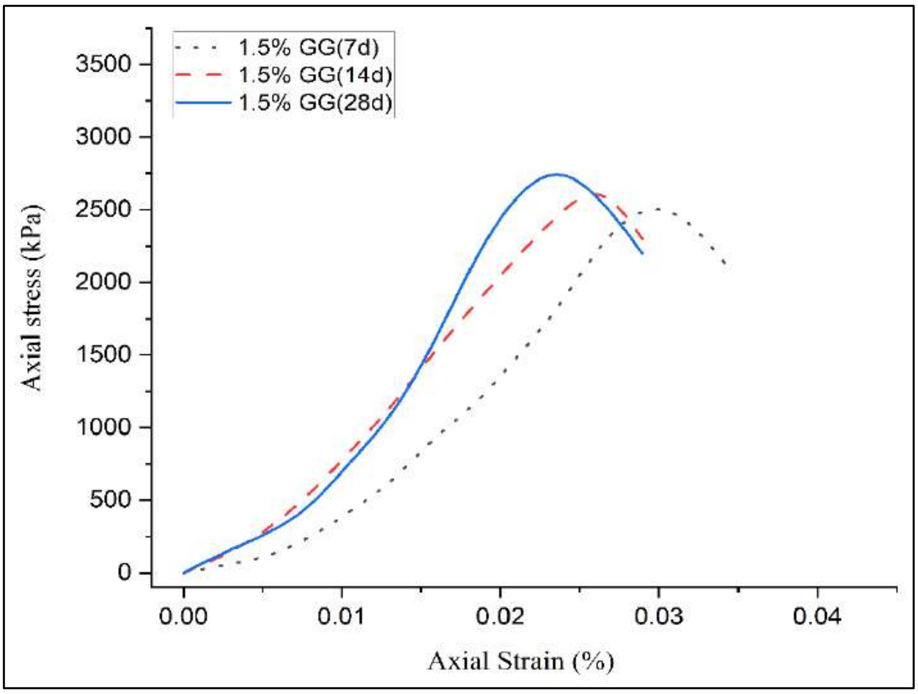


(b)

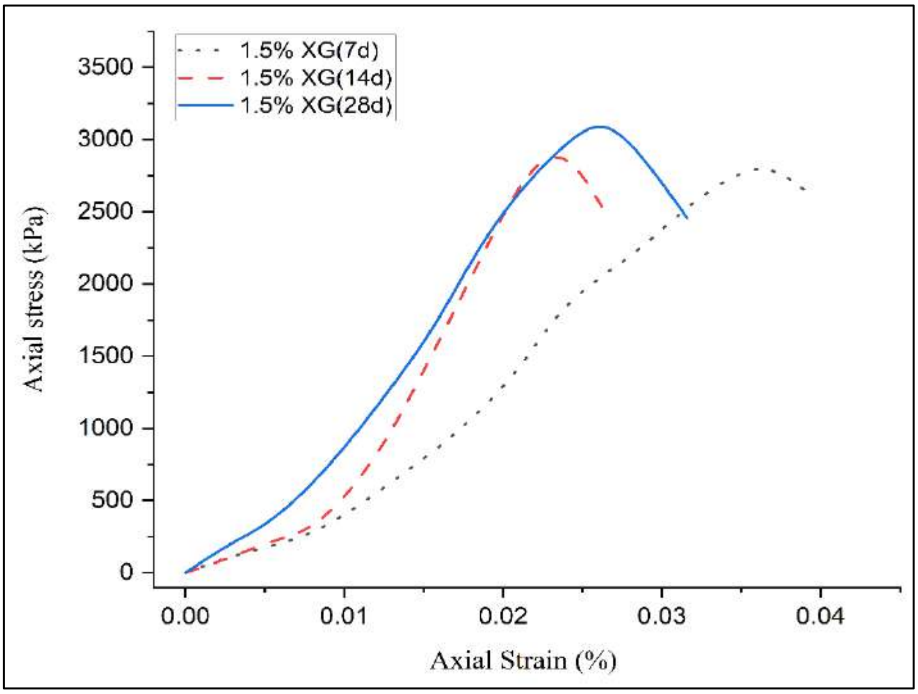


(c)

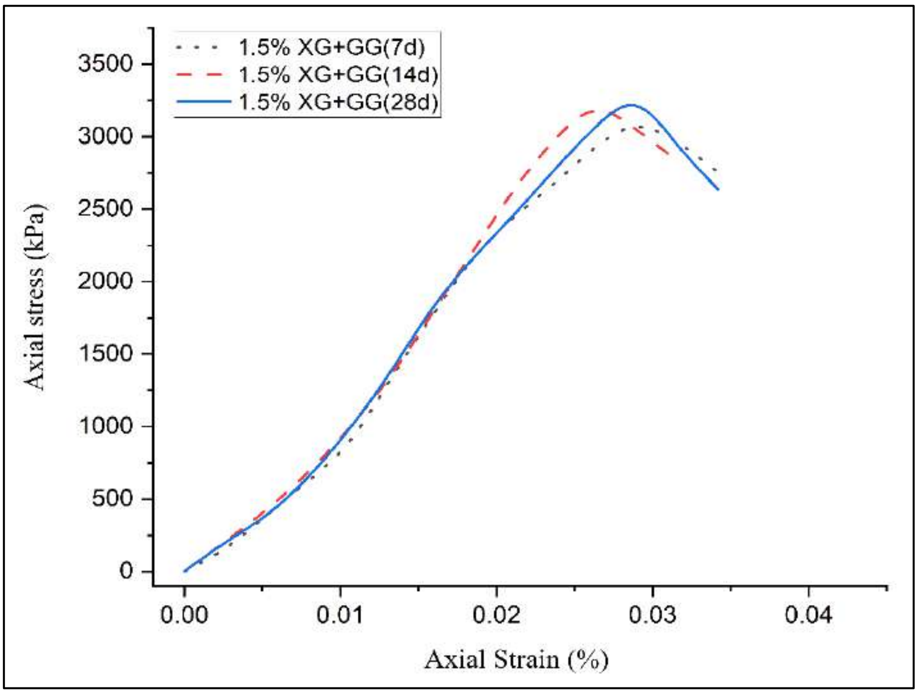
Figure 3.10 Effect of xanthan Gum (XG), guar gum (GG) and composite (XG+GG) on the stiffness of bauxite residue at 28 days curing


1.5% GG(7d)
1.5% GG(14d)
1.5% GG(28d)
Axial stress (kPa)
0
500
1000
1500
2000
2500
3000
3500
0.00
0.01
0.02
0.03
0.04
Axial Strain (%)

(a)


1.5% XG(7d)
1.5% XG(14d)
1.5% XG(28d)
Axial stress (kPa)
0
500
1000
1500
2000
2500
3000
3500
0.00
0.01
0.02
0.03
0.04
Axial Strain (%)

(b)



(c)

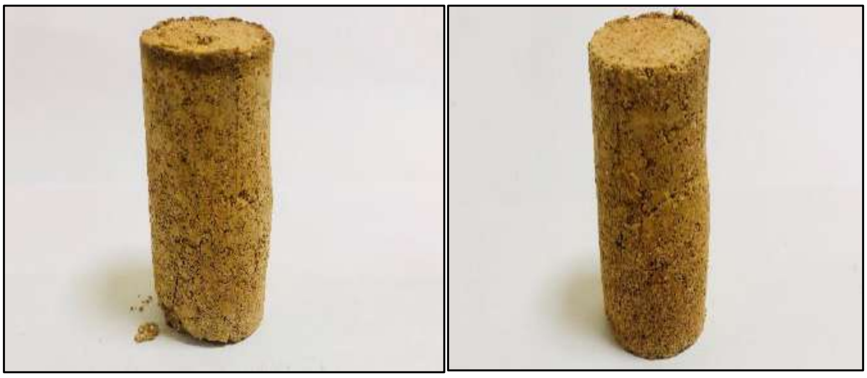
Figure 3.11 Effect of curing period on the stiffness of GG, XG and composite stabilized bauxite residue

3.2.2 Effect of the biopolymer stabilization on Coal mine overburden waste

3.2.2.1 Triaxial compressive test

The triaxial test (unconsolidated–undrained was performed on biopolymer-treated CMO samples at varying concentrations (0.25, 0.5, 1 and 1.5%) and curing time (1 and 7 days)), as per the guidelines of ASTM D 2850. The deviatoric stress-strain responses of the biopolymer-treated CMO samples at different cell pressure are shown in Figures 3.14 to 3.17. The xanthan gum treated CMO sample at 1.5% concentration showed a remarkable increase in cohesion (from 59 to 218 kPa) and friction angle (from 29 to 40°) indicating the inter-particle forces developed by biopolymer addition, which increased with biopolymer concentration and curing time (Figures 3.18 (a) and 3.18 (b)). Guar gum showed better enhancement of strength at 1 day curing time, possibly due to the presence of low water content than the xanthan gum treated samples (Figure 3.19). However, the xanthan gum specimen showed better enhancement of strength after 7 days curing period when the water content in both biopolymer-treated samples was almost nil (approximately 0.5%). This difference is due to guar gum being neutrally charged, and xanthan gum has an electric charge, which results in more reaction of water and soil particles with xanthan gum molecules. Also, the higher plasticity index of soil implies higher undrained shear strength and lower hydraulic conductivity. Therefore, the biopolymer significantly enhances the shear strength and erosion resistance of CMO waste. The sample before and after triaxial test are shown in Figures 3.12 and 3.13 respectively.

Figure 3.12 Sample before triaxial test



(a) (b)

Figure 3.13 Sample after triaxial test (a) 0.5% XG (b) 1% XG

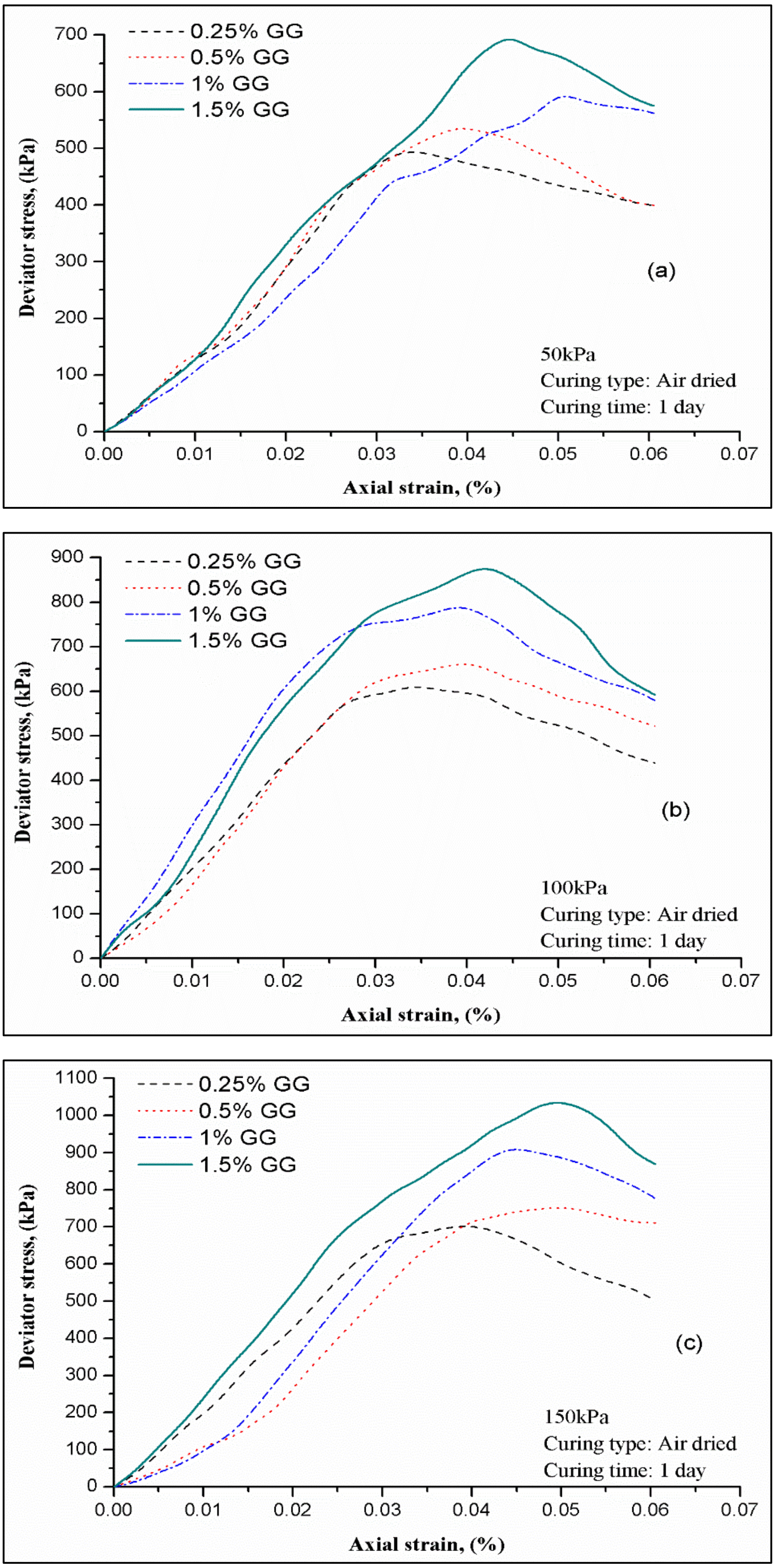


Figure 3.14 A plot of deviator stress and axial strain of GG-treated CMO sample after 1 day curing at different cell pressure (a) 50 kPa, (b) 100 kPa, (c) 150 kPa

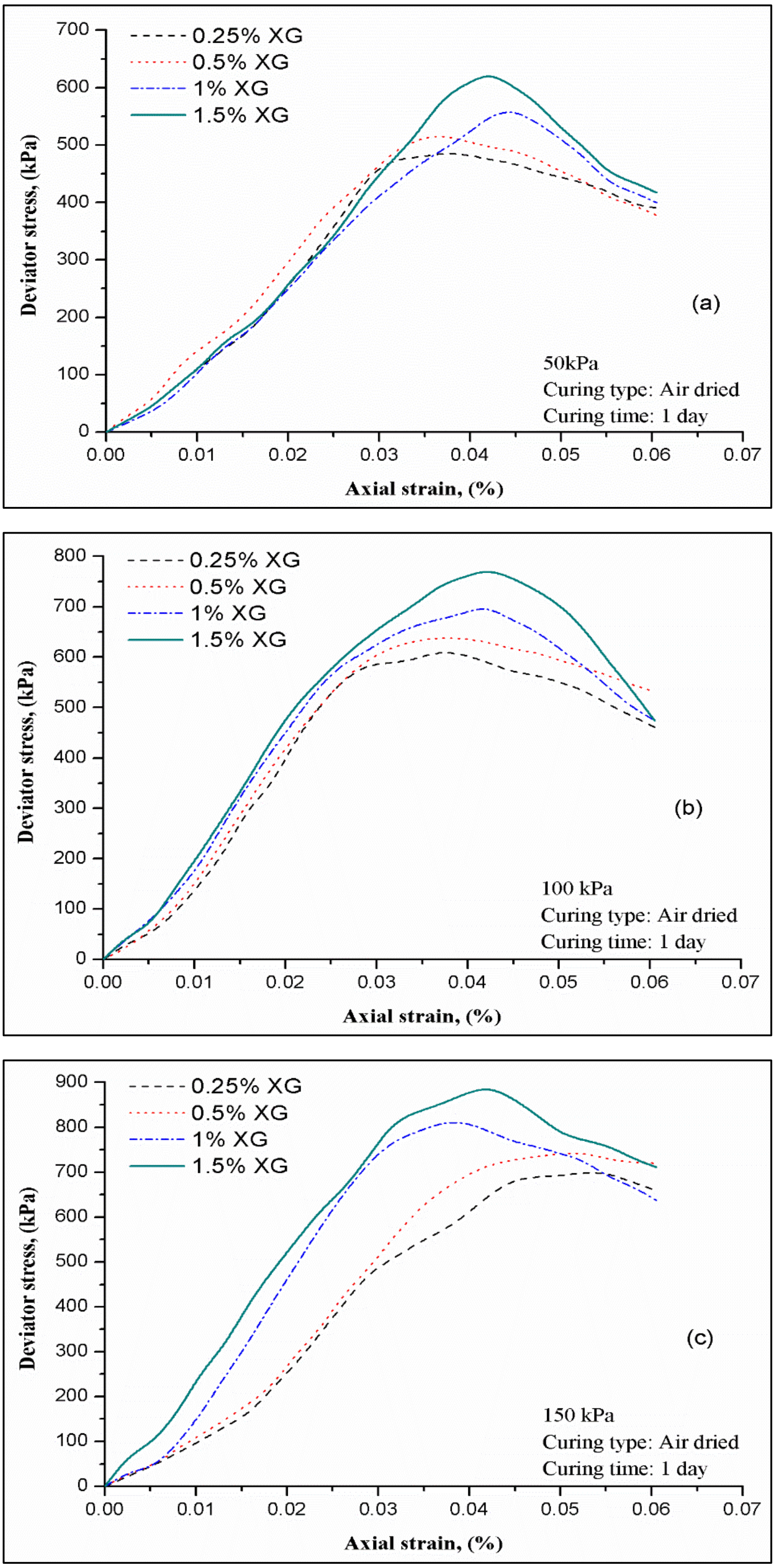


Figure 3.15 A plot of deviator stress and axial strain of XG-treated CMO sample after 1 day curing at different cell pressure (a) 50 kPa, (b) 100 kPa, (c) 150 kPa

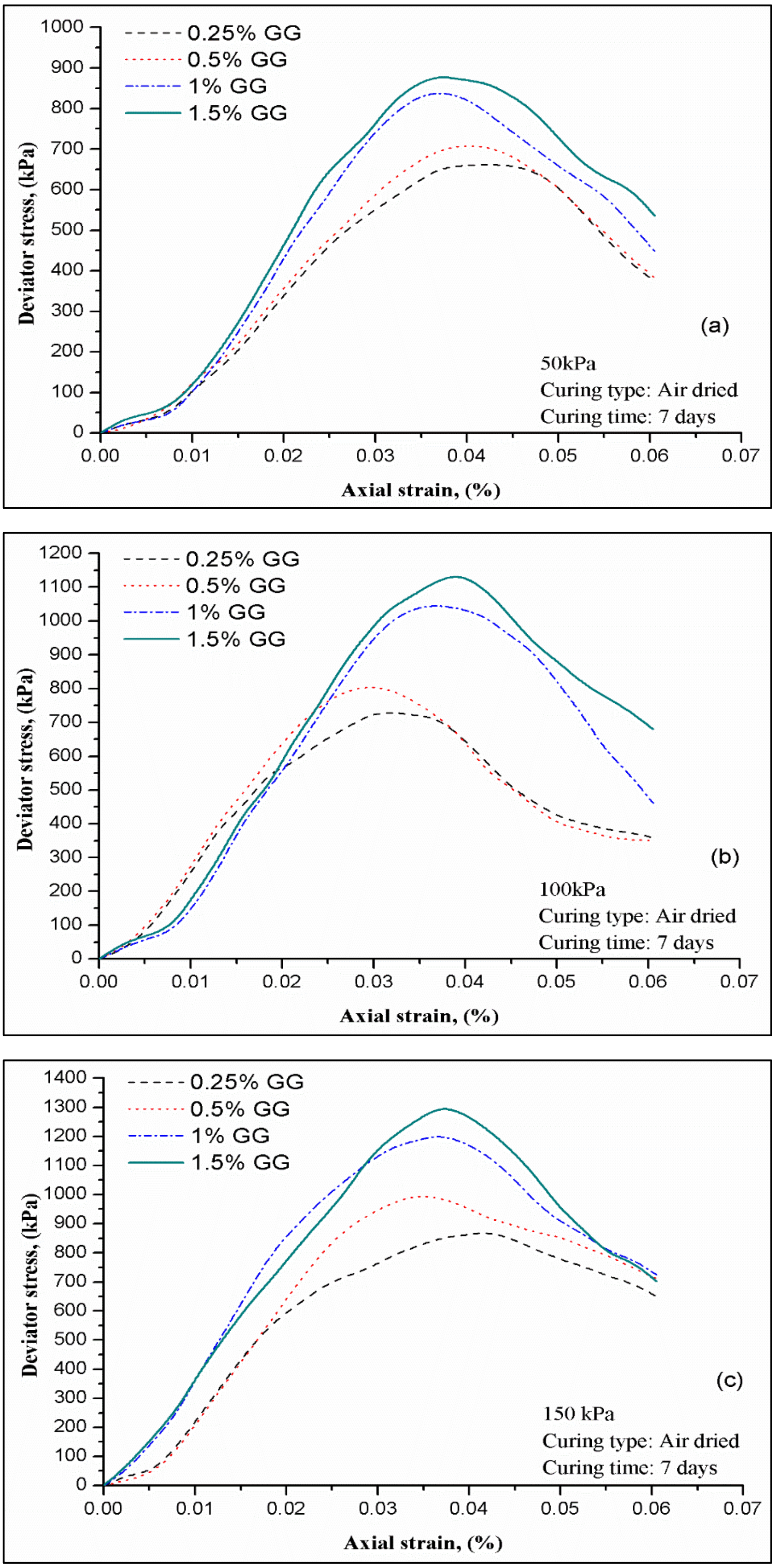


Figure 3.16 A plot of deviator stress and axial strain of GG-treated CMO sample after 7 days curing at different cell pressure (a) 50 kPa, (b) 100 kPa, (c) 150 kPa

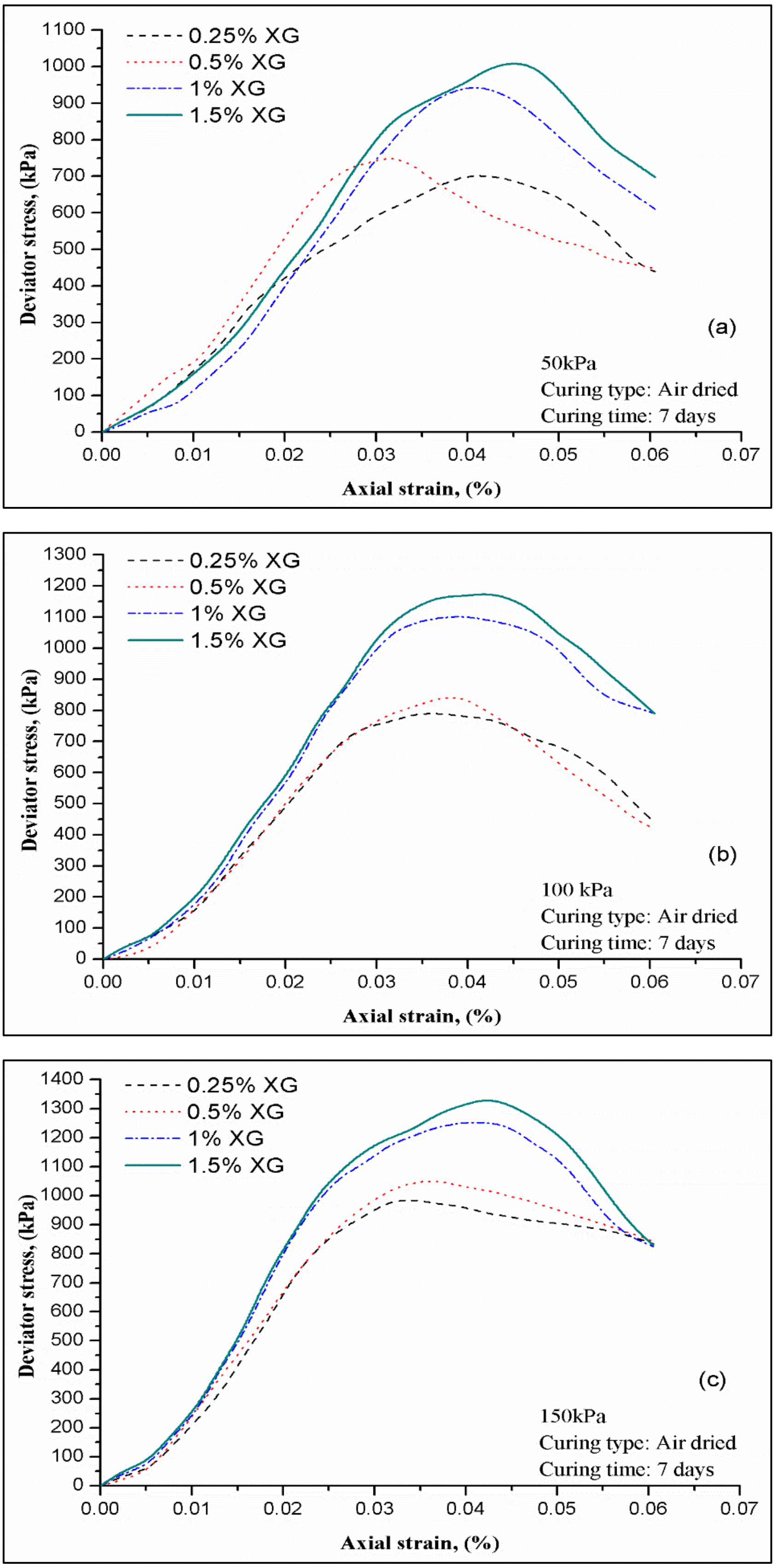
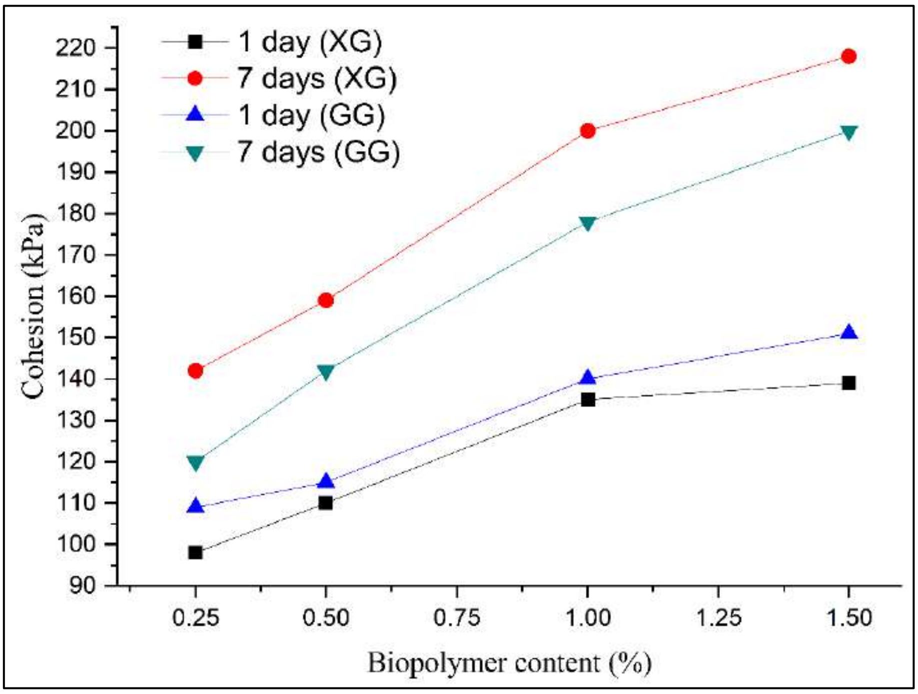
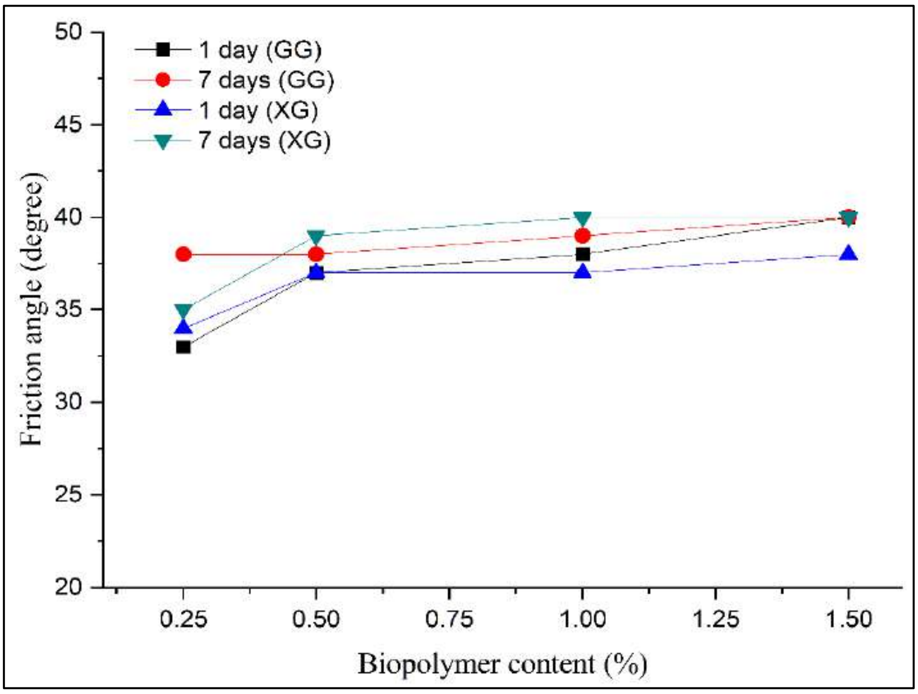


Figure 3.17 A plot of deviator stress and axial strain of XG-treated CMO sample after 7 days curing at different cell pressure (a) 50 kPa, (b) 100 kPa, (c) 150 kPa



(a)



(b)

Figure 3.18 Shear strength parameters of XG and GG-treated CMO sample at different curing periods. (a) Cohesion (kPa), (b) friction angle (deg)

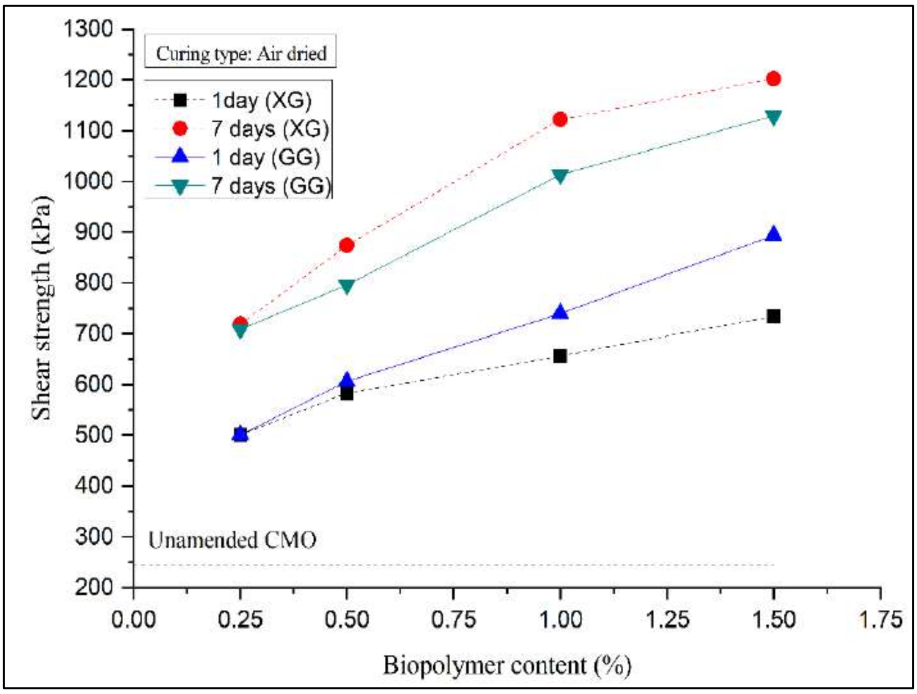
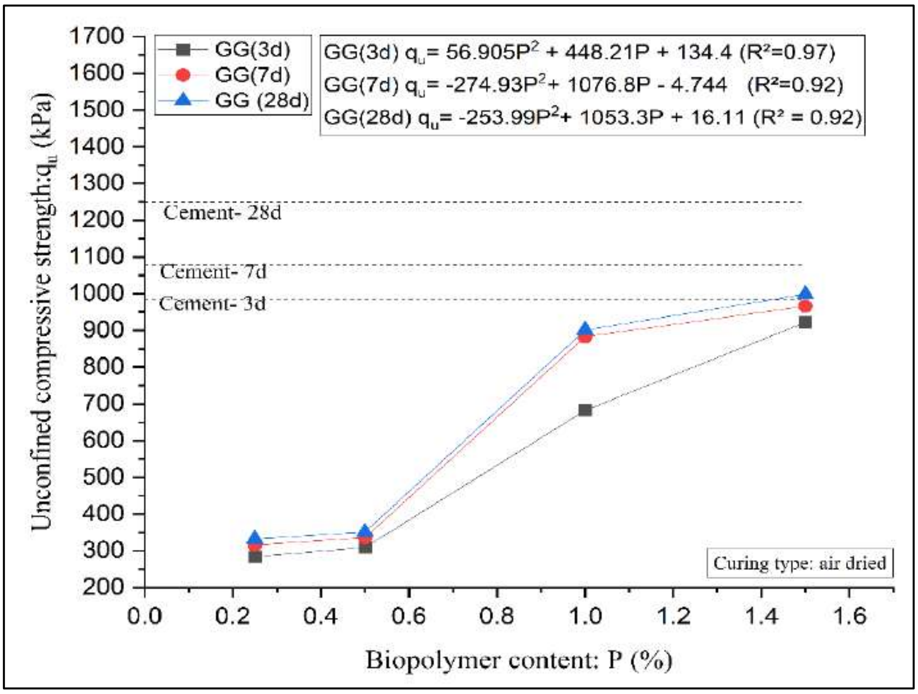


Figure 3.19 Shear strength of CMO treated with different concentrations of XG and GG solution at different curing time

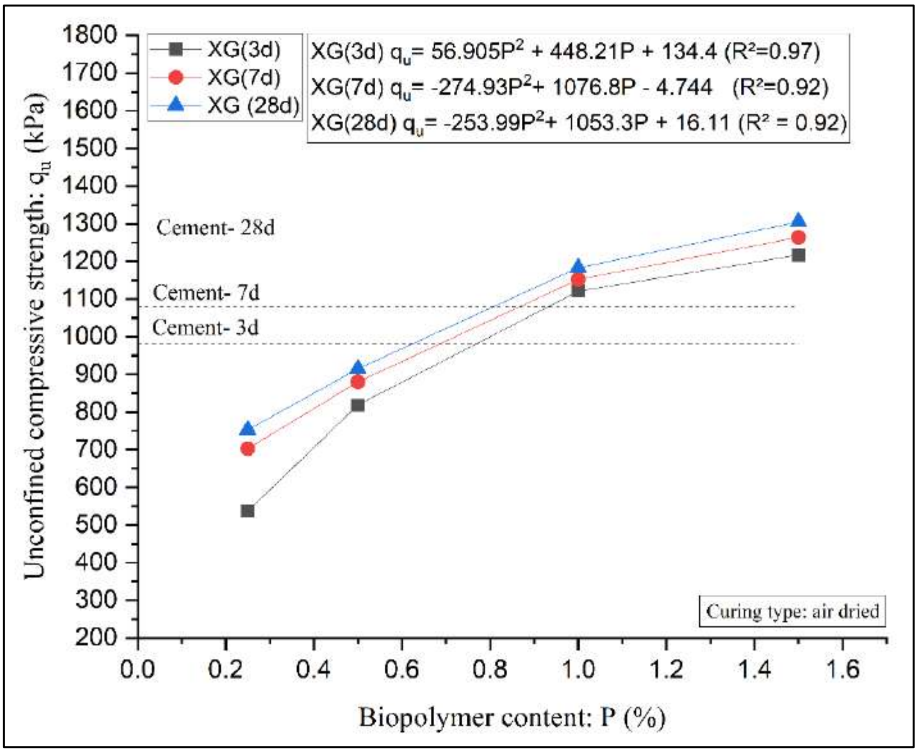
3.2.2.2 Effect of biopolymer on unconfined compressive strength (UCS) of CMO waste

Unconfined compression tests were performed on biopolymer stabilized coal mine overburden waste, and the response was compared with 10% cement-treated CMO plotted in Figure 3.20. The figure shows that the UCS increased with an increase in biopolymer content. In all cases, the UCS of the biopolymer stabilized CMO sample increased significantly compared to untreated CMO waste (due to the negligible UCS of CMO). The result also indicated a moderate increase in UCS with curing time.

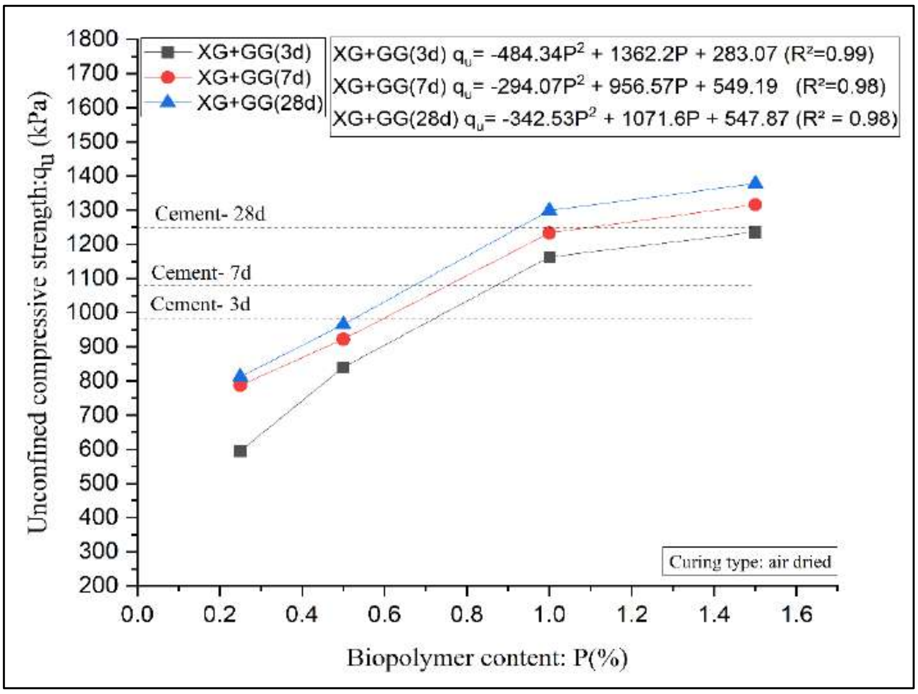
The peak strength of 1% xanthan gum amended CMO at 7 days (1152 kPa on an average) is comparatively higher than the 10 % cement-treated sample at 7 days (1079 kPa on an average). In comparison, guar gum proved ineffective in enhancing shear strength even after curing. The addition of 1.5% of the composite provides a CMO sample with compressive strength higher than the 10% cement-treated sample (40.1, 27.7, and 10.2% higher than cement strength after 3, 7, and 28 days). Also, the effect of XG and its composite on the CMO sample after curing is almost close but much higher than the cohesionless CMO sample.



(a)



(b)



(c)

Figure 3.20 Variation in UCS (q_u) of CMO with Biopolymer and Curing Time (d): (a) Guar Gum, (b) Xanthan Gum, (c) Composite (XG+GG)

The biopolymer treated CMO waste showed higher ductility and significant residual strengthening behavior than 10% cement-treated CMO waste with almost zero residual strength after failure. Many researchers [79, 80] have also reported similar findings. Thus, it appears that the CMO inter-particle strengthening resulted from the formation of thick and dehydrated biopolymer gel among the particles, which supports particle binding even after failure.

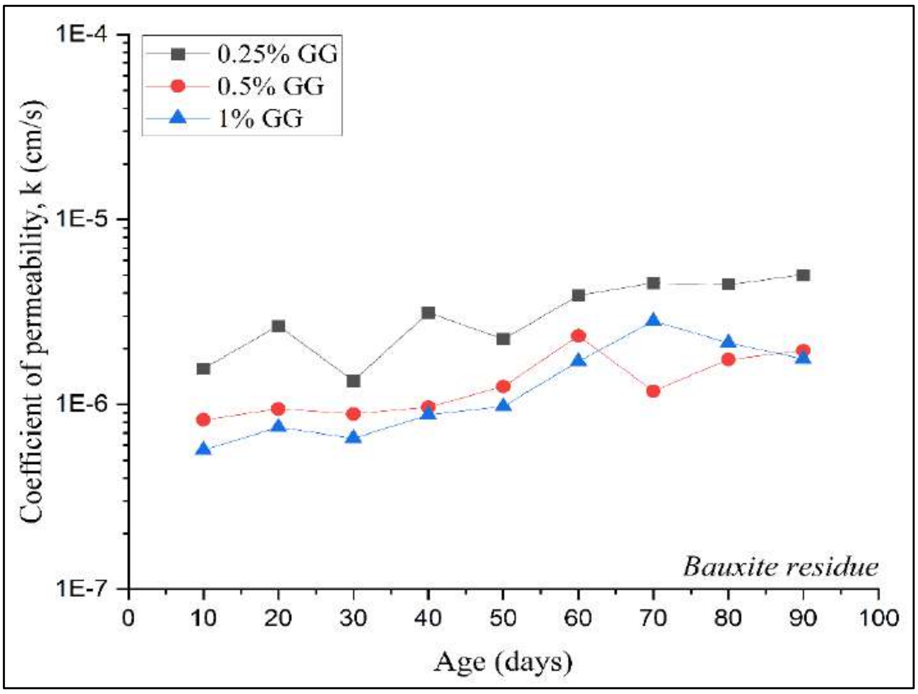
3.3 Permeability Test Analysis

Based on previous studies of permeability test analysis with biopolymers [81-83], an experimental program was developed to study the influence of biopolymer application on the permeability of bauxite residue and coal mine overburden waste.

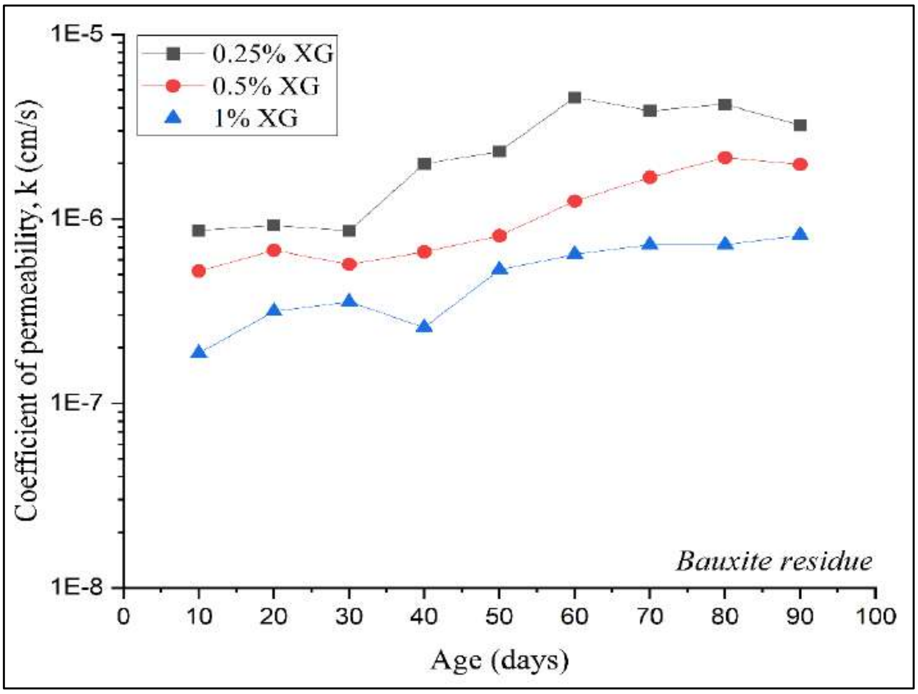
3.3.1 Influence of biopolymer on permeability of bauxite residue

The influence of biopolymer application on bauxite residue permeability was studied by conducting a set of long term falling head permeability tests. Biopolymer solutions (XG, GG, and composite) were prepared according to the method described in section 2.3.6.3 at different concentrations (0.25, 0.5, and 1%). The long term effect of the biopolymer solution on bauxite residue sample was observed for 3 months. First, the biopolymer solution was blended with the required amount of oven-dried bauxite residue sample. Then the mixture was hydrated with water spray and thoroughly mixed with a spatula. Prior to testing, the mixture was kept in a plastic bag for 24 hours to allow inter-particle bonding between biopolymer and bauxite residue particles. Then the samples were compacted into the mould in three layers and left for 24 hours for saturation with water. All the samples were saturated from bottom to top to prevent air bubbles trapping inside the inter-particle voids.

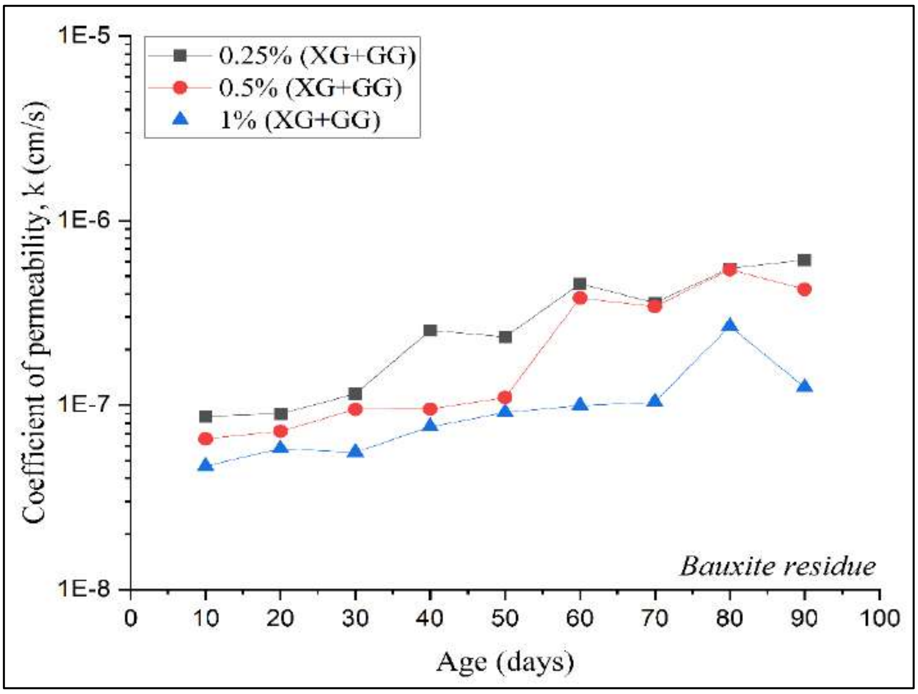
From the results, it is observed that addition of 0.25% xanthan gum and 0.5% guar gum to the bauxite residue sample decreases its hydraulic conductivity by approximately 10 times than the permeability of untreated bauxite residue sample mixed with tap water. Also, addition of 1% composite (XG+GG) gum changes the hydraulic conductivity from $7.65\text{x } 10^{-6}$ to $4.67 \text{ x } 10^{-8}$, which is less than 10^{2} times. When biopolymer is placed in the soil matrix, the cross linking connects the polymeric chains through chemical reactions (due to temperature rise, change in pressure and pH) which rigidifies the whole polymeric structure, enhancing its mechanical strength and reducing permeability. A similar finding has also been reported elsewhere [84]. Hence, the biopolymers have the potential to be applied for hydraulic purpose in geotechnical engineering, such as slurry walls, seepage barriers (temporary) and grouting.


0.25% GG
0.5% GG
1% GG
Coefficient of permeability, k (cm/s)
1E-4
1E-5
1E-6
1E-7
Bauxite residue
0
10
20
30
40
50
60
70
80
90
100
Age (days)

(a)


0.25% XG
0.5% XG
1% XG
Coefficient of permeability, k (cm/s)
1E-5
1E-6
1E-7
1E-8
Bauxite residue
0
10
20
30
40
50
60
70
80
90
100
Age (days)

(b)



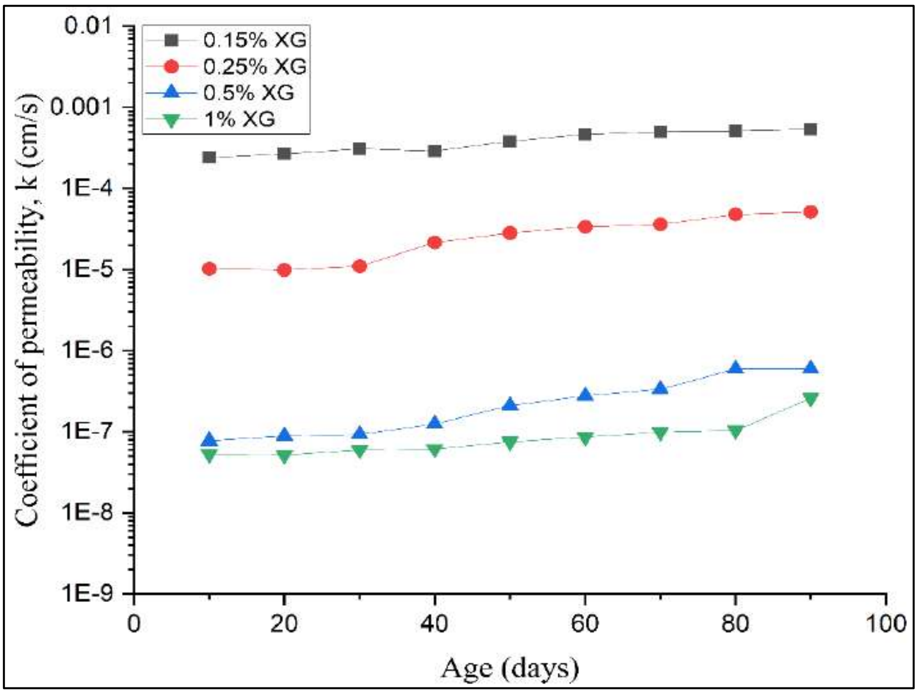
(c)

Figure 3.21 Long term falling head permeability tests on bauxite residue with different concentration of biopolymer solution (0.25, 0.5, and 1%) and 90 days ageing:(a) Guar Gum, (b) Xanthan Gum, and (c) Composite (XG+GG)

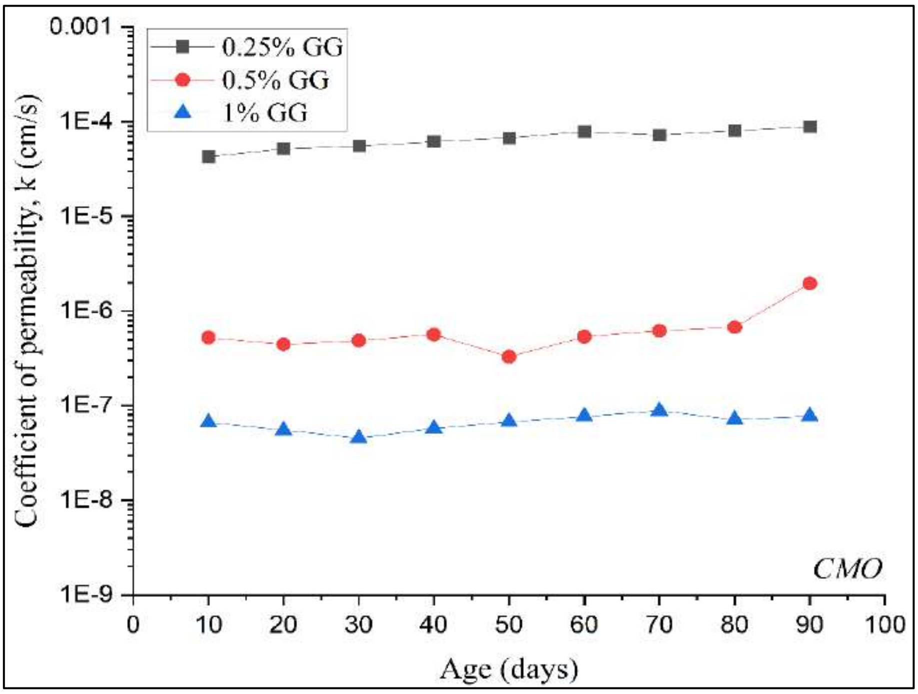
3.3.2 Influence of biopolymer on permeability of Coal mine overburden waste

A long term constant head permeability test was performed to study the influence of biopolymer applications on the highly permeable CMO sample. The samples for the permeability test were prepared by mixing an oven-dried CMO dump with biopolymers. Five mixtures were prepared at different concentrations (0, 0.15, 0.25, 0.5 and 1%) of biopolymers (XG and GG). The different concentrations of biopolymers added were by dry weight of CMO.

Earlier studies have also shown the application of biopolymers on the subsurface soil plugging [85, 86]. It is observed that the hydraulic conductivity decreases with an increase in the concentration of biopolymer. Addition of 0.15% xanthan gum to CMO sample reduces its hydraulic conductivity to almost half of the initial value. However, the influence of 0.15% XG on hydraulic conductivity was not observed after about 60 days, possibly due to the very low content of biopolymer remaining in the sample. The addition of 1% xanthan gum changes the hydraulic conductivity from 5.4×10^{-4} to $5{\cdot}32 \times 10^{-8}$ cm/s, which is less than 10^4 times. As shown in Figure 3.22, the minimum value of permeability for CMO sample is 10^{-8} cm/s. The variation in the permeability might be due to the air bubbles trapped inside the CMO voids. The visible air bubbles in the plastic tube were removed during the experiment, but some air bubbles may have been present inside the CMO sample. At the end of the long-term hydraulic conductivity test, black lenses of slime (approximately 1 mm thick) were observed over the CMO sample, which contained a biopolymer additive (Figure 13(a)). The presence of the slime layer indicates a clogging of voids among CMO particles, hence maintaining the low permeability in the long term. In addition, due to the presence of a biopolymer additive, CMO particles stick to each other, resulting in a linked structure that undergoes a hardening process and becomes rigid (Figure 13(b)). Similar observations for long term permeability test on bonnie silt were also reported by Karimi (1998).



(a)



(b)

Figure 3.22 Long term constant head permeability tests on CMO with different concentration of biopolymer solution (0.25, 0.5, and 1%) and 90 days ageing: (a) Xanthan Gum, (b) Guar gum

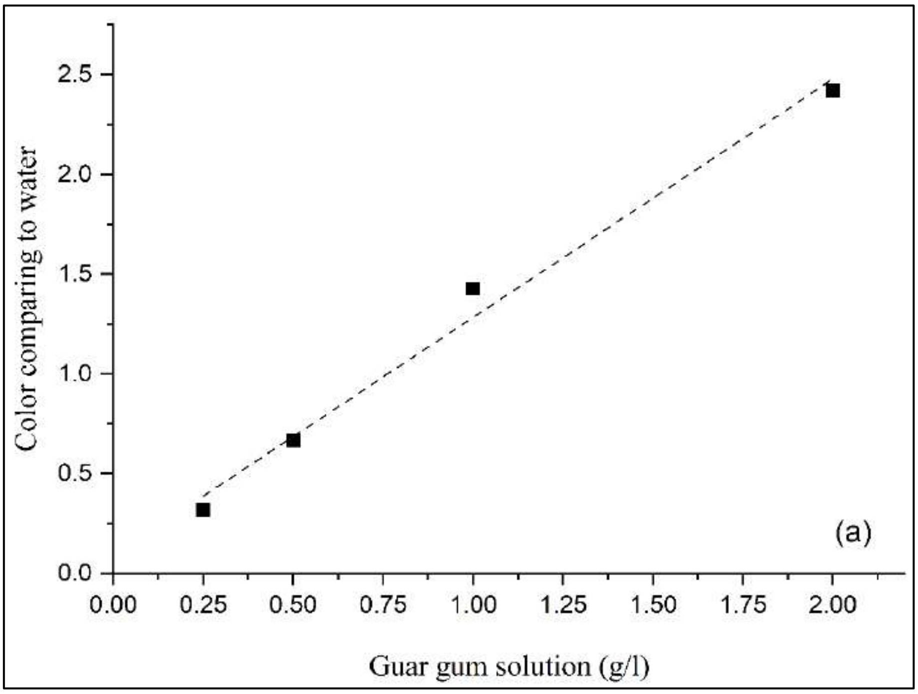
3.4 Total Carbohydrate Method (TCM)

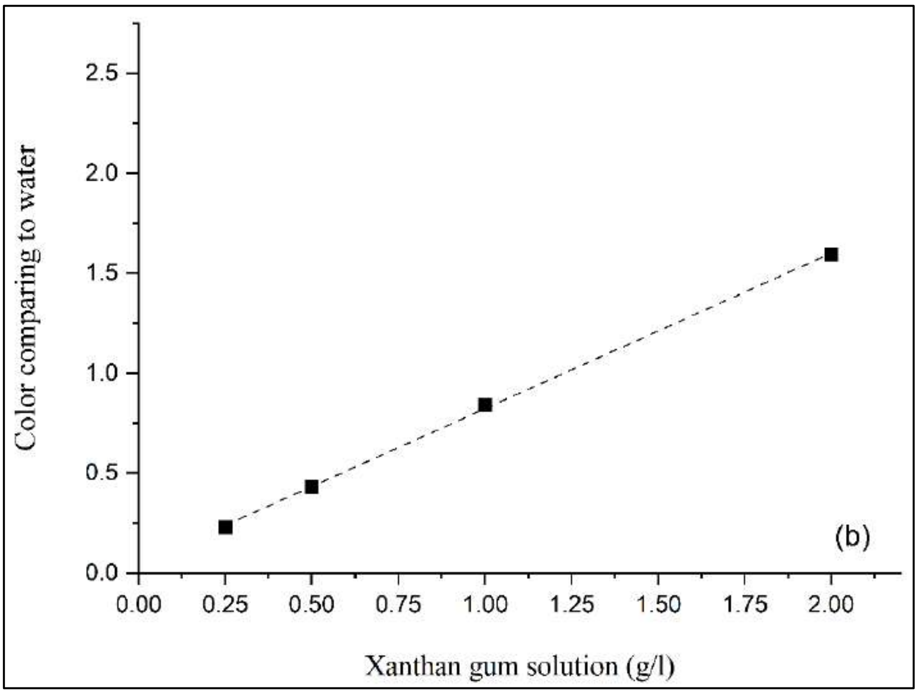
The phenol-sulfuric acid method is an accurate and rapid colorimetric method to determine all classes of carbohydrates, including mono-, di-, oligo-, and polysaccharides. In this method, biopolymer (xanthan and guar gum) is treated with concentrated sulfuric acid, breaking it into sugar units. These free sugar residues then form a complex with the phenol, which is colored and can therefore be determined colorimetrically. This test is designed based on the fact that polysaccharides produce an orange-yellow color when treated with phenol and concentrated sulfuric acid, which can be determined by the colorimetric method [87].The intensity of the orange-yellow color depends on the concentration of the polysaccharides in the solution [88].

3.4.1 Standard line for Biopolymer Solution

The colorimetric method based on phenol/sulfuric acid was followed in the laboratory as outlined below:

A biopolymer solution of 0.1 millilitre was poured into the testing tube along with 0.9 ml of distilled water and 1.0 ml of phenol solution (phenol 5% in distilled water). Next, 5 ml of sulfuric acid was added, and the solution was mixed using a laboratory mixer. Then a chemical reaction takes place, which increases the temperature of the mixture in the test tube. After it gets cool down, the absorbance at 490 nm on spectrophotometer is reported for different concentration of biopolymer (0.25, 0.5, 1, and 2 g per litre) in water. The standard line for different biopolymers (XG, GG, and composite) can be used as a database for detecting the percentage of dissolved biopolymer in water solution (Figure 3.23).





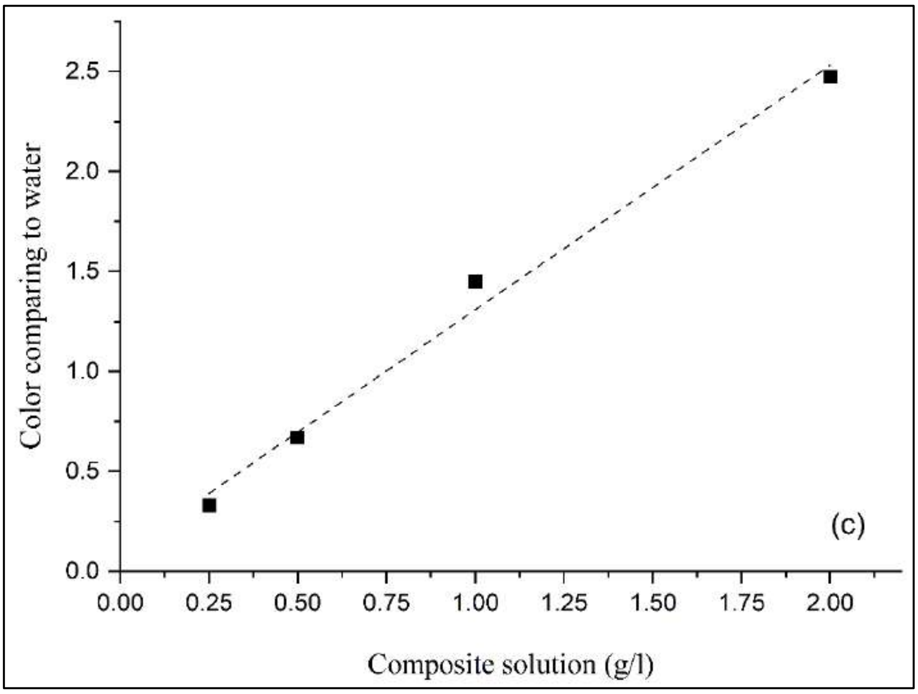


Figure 3.23 Standard line for different biopolymer total carbohydrate test: (a) Guar gum (b) Xanthan gum (c) Composite gum solution

3.4.2 Colorimeter analysis on bauxite residue

A total of 9 bauxite residue samples containing various percentages (0.25, 0.5, and 1%) of biopolymer (XG, GG, composite) were investigated in this research. A falling head permeability test was performed on the sample, and effluent water collected from the permeameter mould was transferred to sealed glass containers. The evaporation of water from the beakers was ignored. The effluent water was collected for approximately two months from each sample and was analyzed using TCM, and readings were reported through the colorimeter method. The collected effluent water from each sample was analyzed using the total carbohydrate method, and the readings in the colorimeter were recorded (Table 3.2). The concentration of corresponding gum in the effluent water was estimated by comparing the colorimeter readings with the standard line of respective gum (Figure 3.23).

Table 3.2 Results of bauxite residue effluent water colorimeter analysis

No.	Biopolymer with Bauxite residue sample	Reading in colorimeter	Concentration of biopolymer in effluent water (g/l)
1.	Bauxite residue sample with 0.25% GG	0.144	< 0.25
2.	Bauxite residue sample with 0.5% GG	0.187	< 0.25
3.	Bauxite residue sample with 1% GG	0.210	< 0.25
4.	Bauxite residue sample with 0.25% XG	0.138	< 0.25
5.	Bauxite residue sample with 0.5% XG	0.162	< 0.25

6.	Bauxite residue sample with 1% XG	0.220	< 0.25
7.	Bauxite residue sample with 0.25 % XG+GG	0.149	< 0.25
8.	Bauxite residue sample with 0.5 % XG+GG	0.175	< 0.25
9.	Bauxite residue sample with 1 % XG+GG	0.231	< 0.25

From the above results, it can be concluded that the concentration of all the biopolymers in the effluent water was less than 0.25g/l, indicating a large percentage of biopolymers (XG, GG, and XG+GG) had been retained in the bauxite residue sample. However, the mechanism of retention is unclear and still needs to be explored. Following the same procedure, Karimi [77] also reported a large percentage of xanthan gum retention in the silver sand particles after the effluent water collection for one week.

3.4.3 Colorimeter analysis on Coal mine overburden waste

A long-term constant head permeability test was performed on the set of 7 samples of CMO, and the effluent water was collected to determine the corresponding concentration of gum (XG and GG). Since CMO has high permeability, a period of 1 month to collect the effluent was considered satisfactory. The colorimeter readings were recorded using the total carbohydrate method (Table 3.3) and compared with the standard line in Figure 3.23.

Table 3.3 Results of Coal mine overburden waste effluent water colorimeter analysis

No.	**Biopolymer with CMO sample**	**Reading in colorimeter**	**Concentration of biopolymer in effluent water (g/l)**
1.	CMO sample with 0.15% XG	0.152	< 0.25
2.	CMO sample with 0.25% XG	0.184	< 0.25
3.	CMO sample with 0.5 % XG	0.252	< 0.25
4.	CMO sample with 1 % XG	0.266	< 0.25
5.	CMO sample with 0.25 % GG	0.167	< 0.25
6.	CMO sample with 0.5 % GG	0.185	< 0.25
7.	CMO sample with 1 % GG	0.210	< 0.25

The colorimeter readings show that the biopolymer concentration in the effluent water of the CMO sample was less than 0.25g/l, indicating the retention of the large concentration of biopolymer (XG and GG) in the sample. Also, CMO is highly permeable due to large pore volume; it was expected that percentage of biopolymer in effluent water would be high. However, colorimeter readings indicated that a large percentage of gum was retained inside the CMO sample. It appears that other adsorption parameters, rather than retention in the pores, have played a role in retaining gum inside the CMO sample.

3.5 Durability Study (Freezing and thawing test)

Following the ASTM D560M-15 standard, the freezing and thawing resistance of the stabilized biopolymer bauxite residue samples were tested. This test method is used to determine the resistance of compacted biopolymer stabilized bauxite residue matrix to repeated cycles (10 cycles) of freezing and thawing (F-T) and determine the optimum composition required to achieve a degree of adequate hardness to resist field weathering. To assess the durability of biopolymer stabilized bauxite residue, strength analysis with and without thermal treatment subjected to 0, 1, 3, 5, 8, and 10 F-T cycles was conducted and discussed subsequently.

3.5.1 Sample preparation using Thermo-gelation

The bauxite residue sample was supplied from the disposal field to the laboratory; before testing, the residue was oven-dried, sieved, and grinded to remove any larger particles. To prepare the blend of biopolymer treated bauxite residue mixtures, the required amount of dry biopolymer powder was dissolved in hot distilled water (100 °C) to obtain a solution at varying (m_b /m_w) concentrations. Then, the biopolymer solution at desired concentration was mixed using a temperature-controlled magnetic stirrer (REMI). Finally, oven heated bauxite residue (80 ºC) was quickly mixed on a hot plate with a prepared solution to maintain the temperature of the biopolymer-bauxite residue mixture. During the thermal treatment process, the continuous mixing and heating of the biopolymer solution allow the polymeric chains to scatter and form a hydrocolloid solution. Then, the polymeric chain twist with other chains or molecules as the solution cools and forms a hydrogen bonding to develop a rectangular matrix [89].

The prepared specimens were then wrapped in a sealed plastic bag and cured for 7, 14, and 28 days for F-T analysis. The curing of the samples results in the advancement of cross-linking of the particles, which rigidifies the polymer structure by connecting the polymeric chains through chemical chains (i.e., hydrogen bonding). The loss of moisture content in all the specimens was observed to be lower than 0.25%.

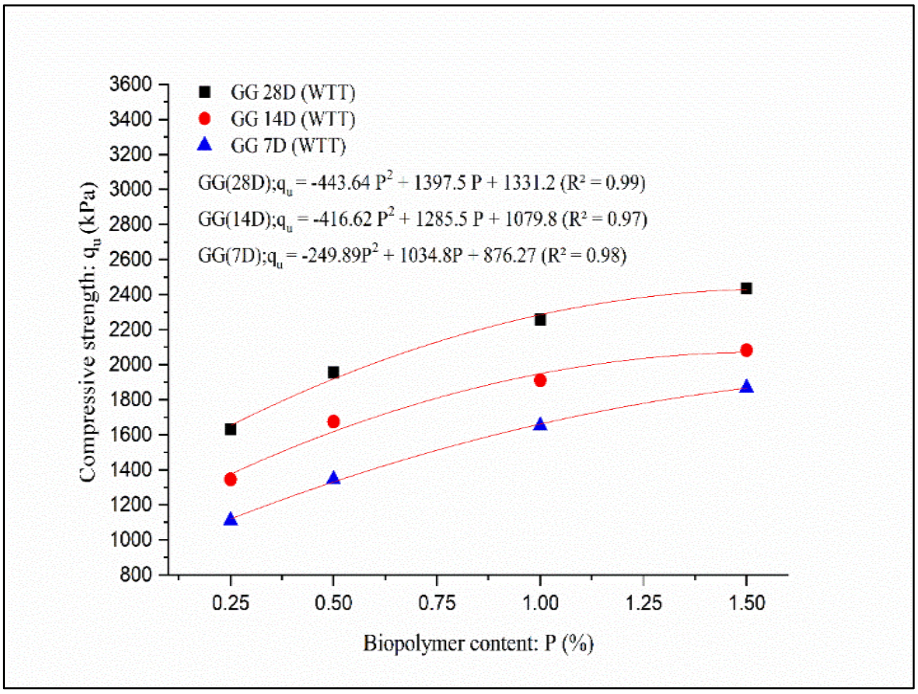
3.5.2 Sample preparation without Thermal treatment

Biopolymer-bauxite residue mixed samples were also prepared without thermal treatment to compare their impact towards F-T resistance. Both biopolymers (XG and GG) solutions were individually mixed with bauxite residue at room temperature (25 ± 5 ºC) without any heat source for preparing the samples.

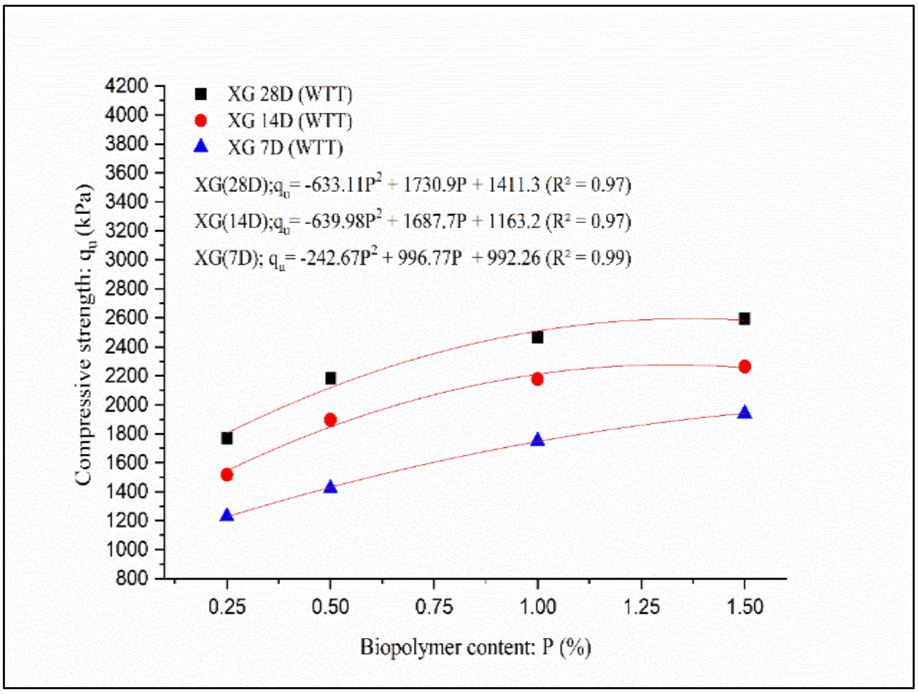
3.5.3 Effect of Thermo-gelation on Compressive Strength before F-T process

The compressive strength of all the biopolymer treated bauxite residue samples (with thermal treatment (TT) and without thermal treatment (WTT)) are presented in Figure 3.24. After curing for 7, 14, and 28 days, it is observed that there is a considerable increase in stiffness with increasing biopolymer concentration. The compressive strength of untreated bauxite residue samples is 247 kPa (no curing), when blended with 1.5% XG and without thermal treatment, showed significant UCS values of 1940, 2265, and 2596 kPa at 7, 14, and 28 days curing time respectively. Meanwhile, a higher strengthening effect (2289, 2501, and 2832 kPa) was observed for samples prepared with the thermal treatment for the same curing period. For samples at 28 days curing period, the thermo-gelation produces a denser structure of bauxite residue particles due to the formation of enhanced inter-particle bonding. Thus, resulting in a better enhancement of the strength compared to samples prepared without thermal treatment.

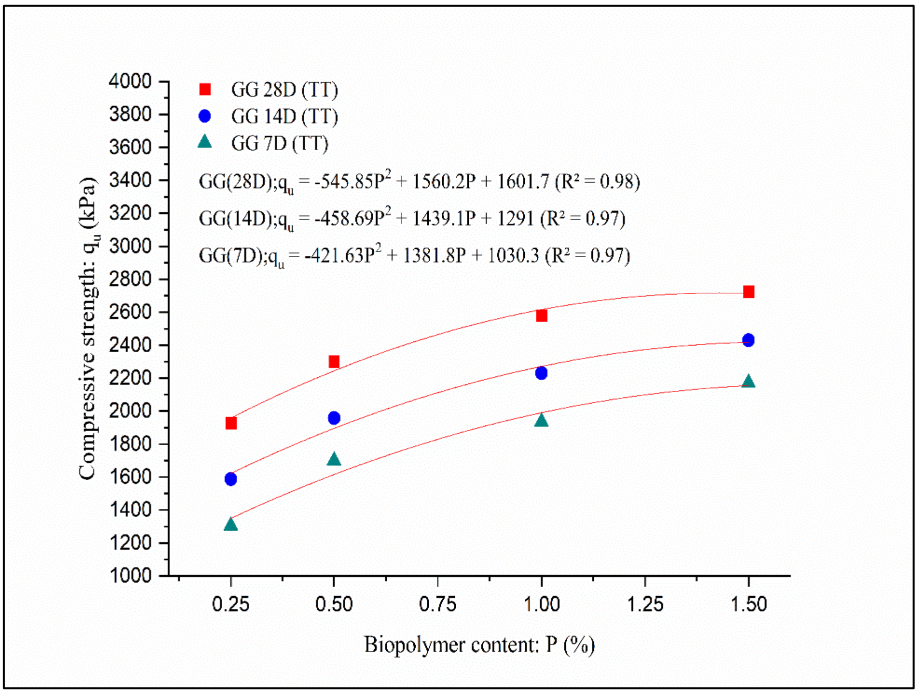
Numerous studies in the past have also validated that biopolymer can form a cross-linking network (double helix hydrogel) within the structure which also enhances the rigidity and stability with a longer curing time [84, 90]. The effect of curing time on the compressive strength of the stabilized bauxite residue samples showed enhanced strength indicating the influence of curing time required for the formation of hydrogen bonds and/or ionic bonds to strengthen the stabilized sample. Many researchers have reported similar observations [89, 91].



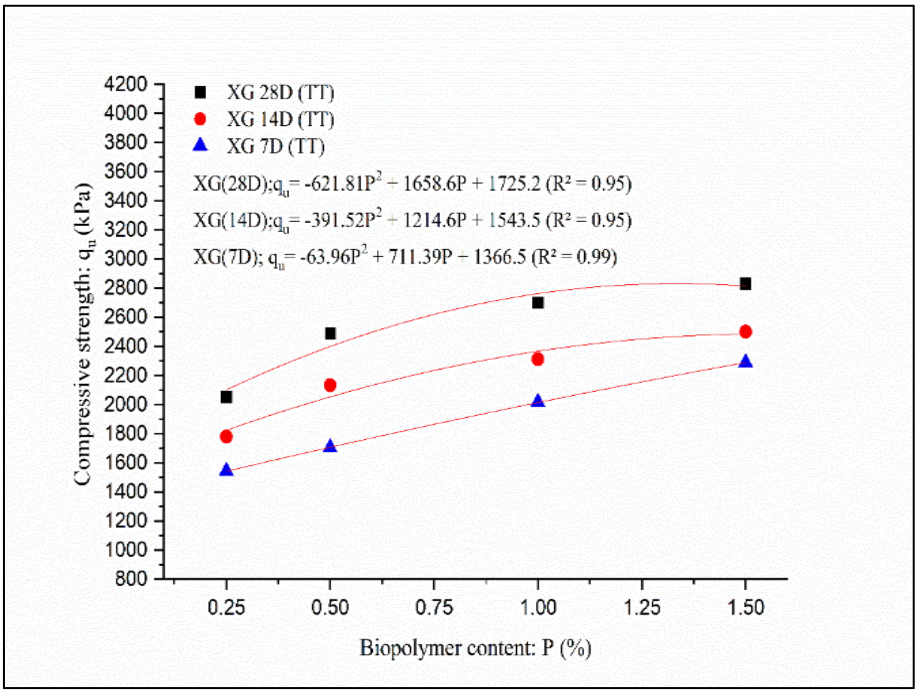
(a)



(b)



(c)



(d)

Figure 3.24 Variation in UCS (q_u) with varying biopolymer concentration and curing time: 7 days (7D), 14 days (14D) and 28 days (28D): (a) GG(WTT), (b) XG(WTT), (c) GG(TT), and (d) XG(TT)

3.5.4 Effect of biopolymer on freezing and thawing resistance

To examine the impact of biopolymer on F-T resistance, the untreated and stabilized (with and without thermal treatment procedure) bauxite residue samples were exposed to F-T (0, 1, 3, 5, 8, and 10) cycles and then tested for UCS to evaluate the loss in strength. The effect of F-T cycles on the compressive strength of the untreated and stabilized bauxite residue samples are shown in Figures 3.25, 3.26, and 3.27, respectively. The results show that for thermally treated bauxite residue samples containing 1.5% XG and subjected to 10 F-T cycles, the percentage loss in unconfined compressive strength, at 7, 14, and 28 days curing periods decreased from 46.0% (untreated bauxite residue) to 13.1, 12.5, and 11.5% respectively. The variation in compressive strength with F-T exposure is due to the particle rearrangements and initiation of micro-cracks within the sample structure that causes cracking and structural deformation, resulting in a loss of stiffness and strength. Many researchers have reported similar findings [92-94].

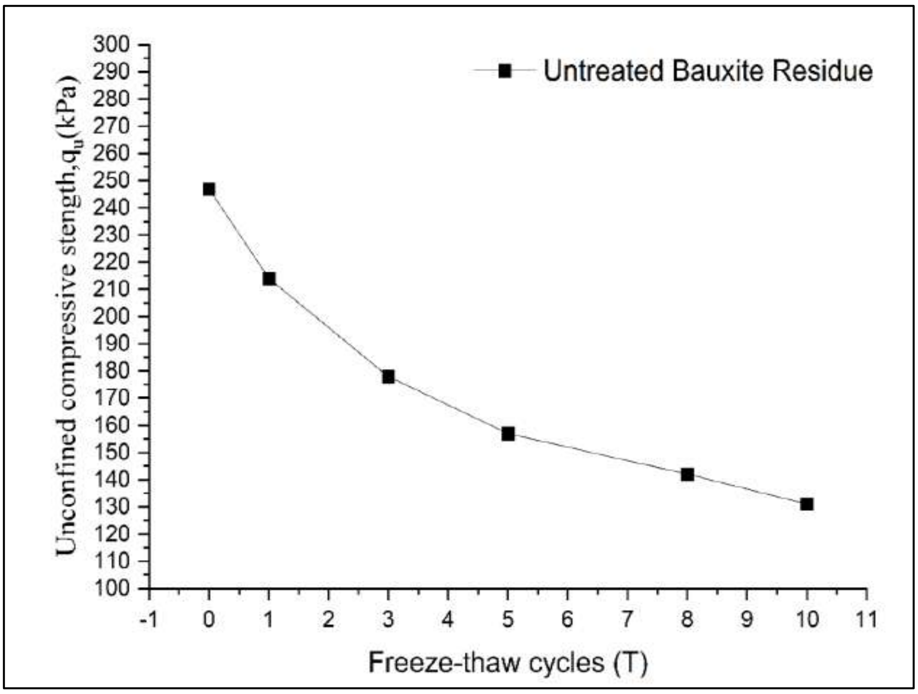
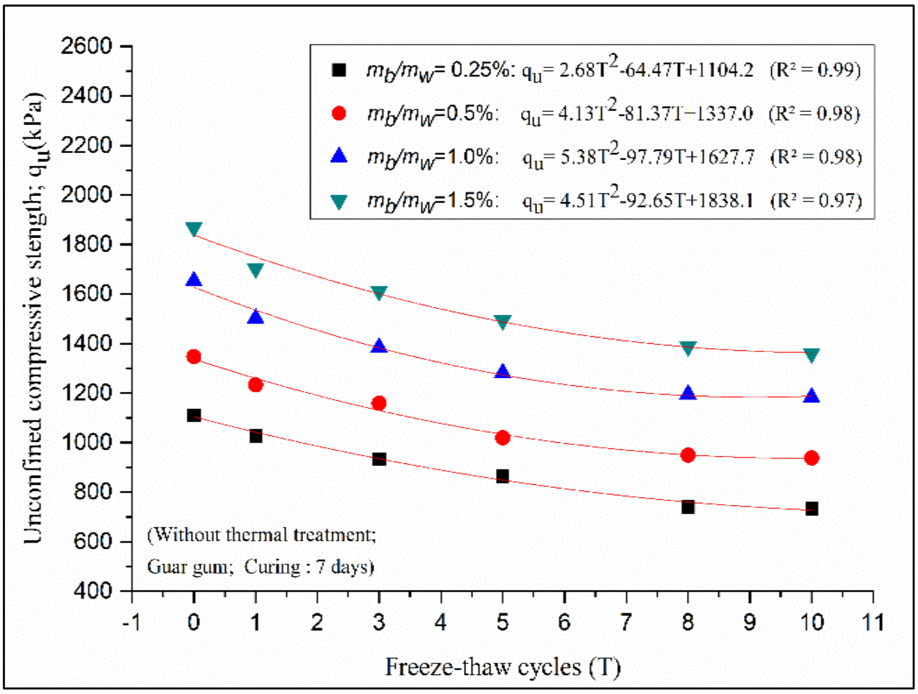
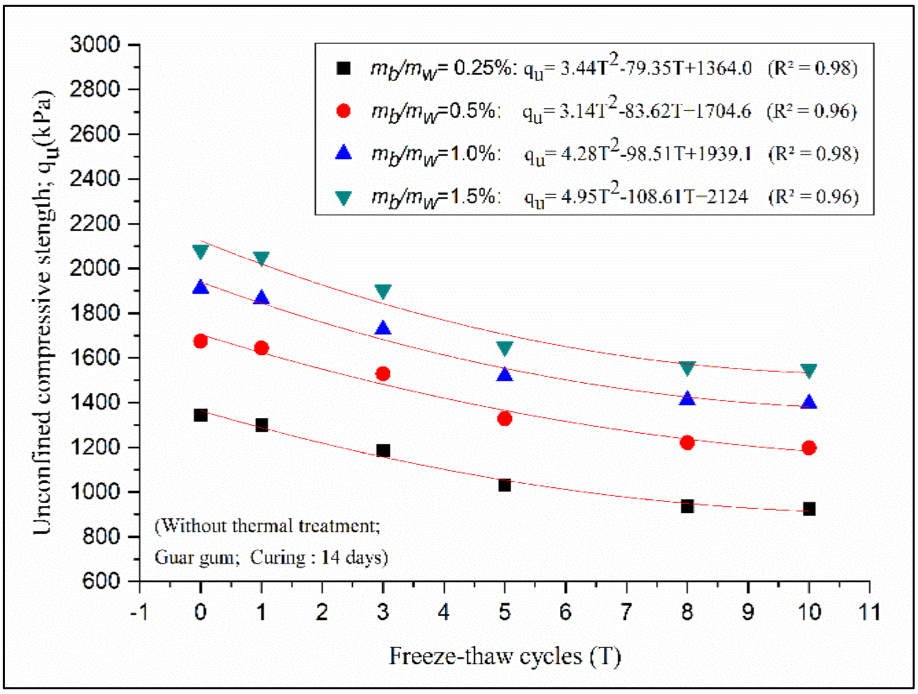


Figure 3.25 Effect of F-T cycles on the UCS (qu) of the untreated bauxite residue

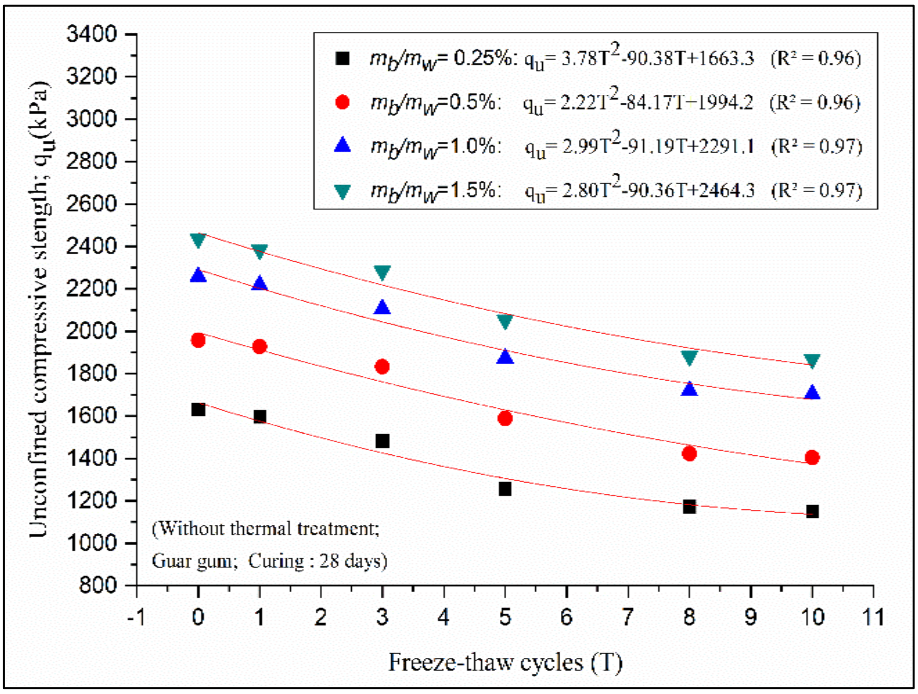
It is evident from Figures 3.26 and 3.27 that the biopolymer stabilized bauxite residue samples (with and without thermal treatment) exhibited high resistance to the F-T process. After 10 F-T cycles, the stabilized bauxite residue samples prepared without thermal treatment (Figure 4) and cured for 7, 14, and 28 days, the loss in compressive strength was recorded as 21.9, 20.3, and 19.7% (1.5% XG) compared to 27.2, 25.6, and 23.3% (1.5% GG) respectively. The experimental results of the UCS test on biopolymer stabilized bauxite residue samples reveal its significant role in durability after F-T cycles. There is a decrease in the UCS values for all mixtures (untreated and treated bauxite residue samples) with an increase in freezing and thawing cycles.



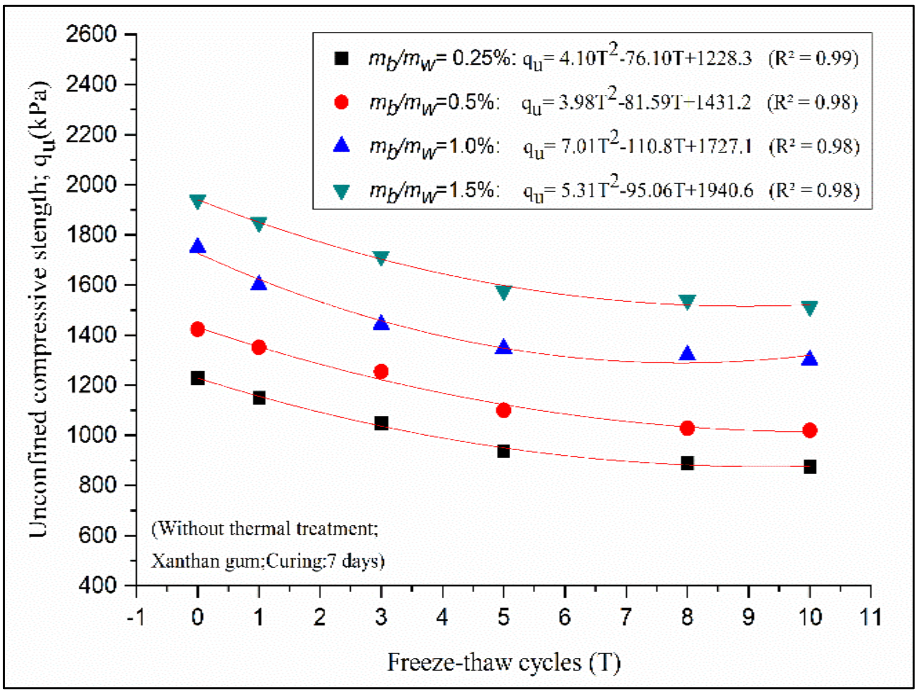
(a)


■ m_b/m_w= 0.25%: $q_u = 3.44T^2 - 79.35T + 1364.0$ ($R^2 = 0.98$)
● m_b/m_w=0.5%: $q_u = 3.14T^2 - 83.62T + 1704.6$ ($R^2 = 0.96$)
▲ m_b/m_w=1.0%: $q_u = 4.28T^2 - 98.51T + 1939.1$ ($R^2 = 0.98$)
▼ m_b/m_w=1.5%: $q_u = 4.95T^2 - 108.61T + 2124$ ($R^2 = 0.96$)
(Without thermal treatment;
Guar gum; Curing : 14 days)
Unconfined compressive stength; q_u(kPa)
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)

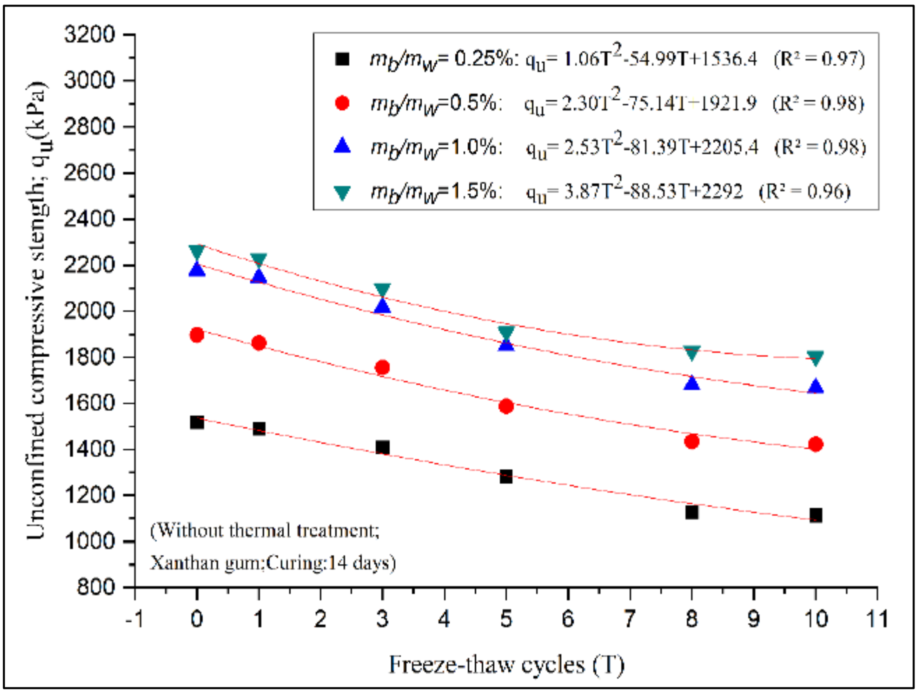
(b)


■ m_b/m_w= 0.25%: $q_u = 3.78T^2 - 90.38T + 1663.3$ ($R^2 = 0.96$)
● m_b/m_w=0.5%: $q_u = 2.22T^2 - 84.17T + 1994.2$ ($R^2 = 0.96$)
▲ m_b/m_w=1.0%: $q_u = 2.99T^2 - 91.19T + 2291.1$ ($R^2 = 0.97$)
▼ m_b/m_w=1.5%: $q_u = 2.80T^2 - 90.36T + 2464.3$ ($R^2 = 0.97$)
(Without thermal treatment;
Guar gum; Curing : 28 days)
Unconfined compressive stength; q_u(kPa)
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)

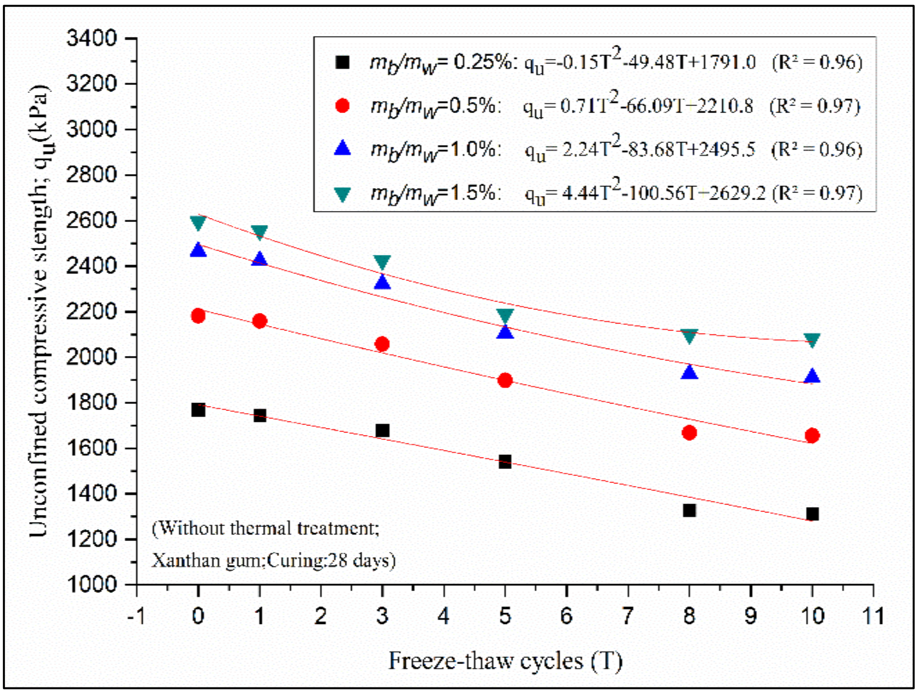
(c)


Unconfined compressive stength; q_u(kPa)
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)
m_b/m_w= 0.25%: q_u= 4.10T^2-76.10T+1228.3 (R² = 0.99)
m_b/m_w=0.5%: q_u= 3.98T^2-81.59T+1431.2 (R² = 0.98)
m_b/m_w=1.0%: q_u= 7.01T^2-110.8T+1727.1 (R² = 0.98)
m_b/m_w=1.5%: q_u= 5.31T^2-95.06T+1940.6 (R² = 0.98)
(Without thermal treatment;
Xanthan gum;Curing:7 days)

(d)

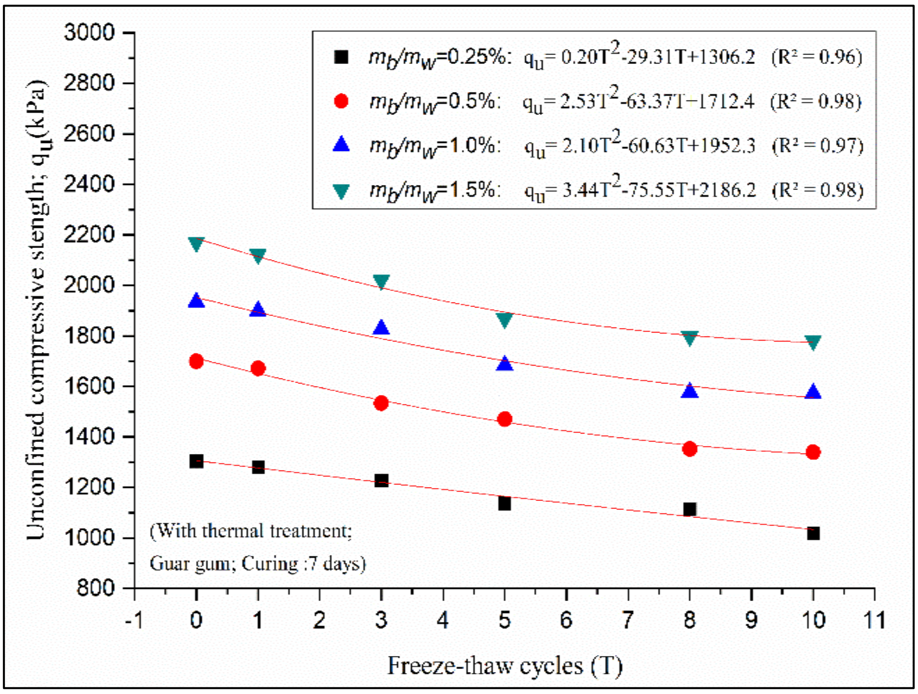

Unconfined compressive stength; q_u(kPa)
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)
m_b/m_w= 0.25%: q_u= 1.06T^2-54.99T+1536.4 (R² = 0.97)
m_b/m_w=0.5%: q_u= 2.30T^2-75.14T+1921.9 (R² = 0.98)
m_b/m_w=1.0%: q_u= 2.53T^2-81.39T+2205.4 (R² = 0.98)
m_b/m_w=1.5%: q_u= 3.87T^2-88.53T+2292 (R² = 0.96)
(Without thermal treatment;
Xanthan gum;Curing:14 days)

(e)

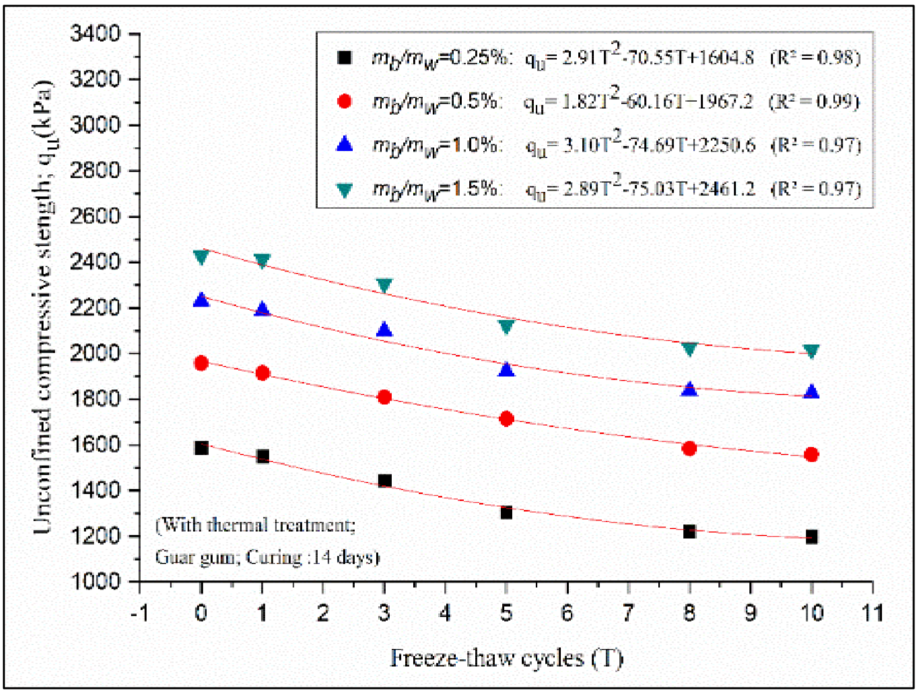


(f)

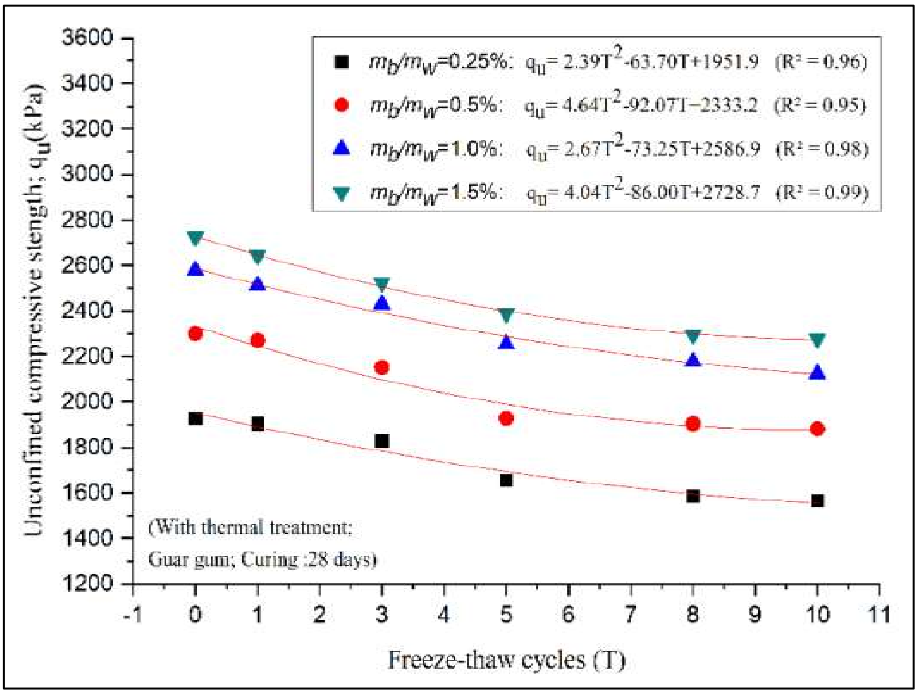
Figure 3.26 Effect of F-T cycles on stabilized bauxite residue without thermal treatment at varying biopolymer concentration and curing time: (a) GG(7D), (b) GG(14D), (c) GG(28D), (d) XG(7D), (e) XG(14D), and (f) XG(28D)



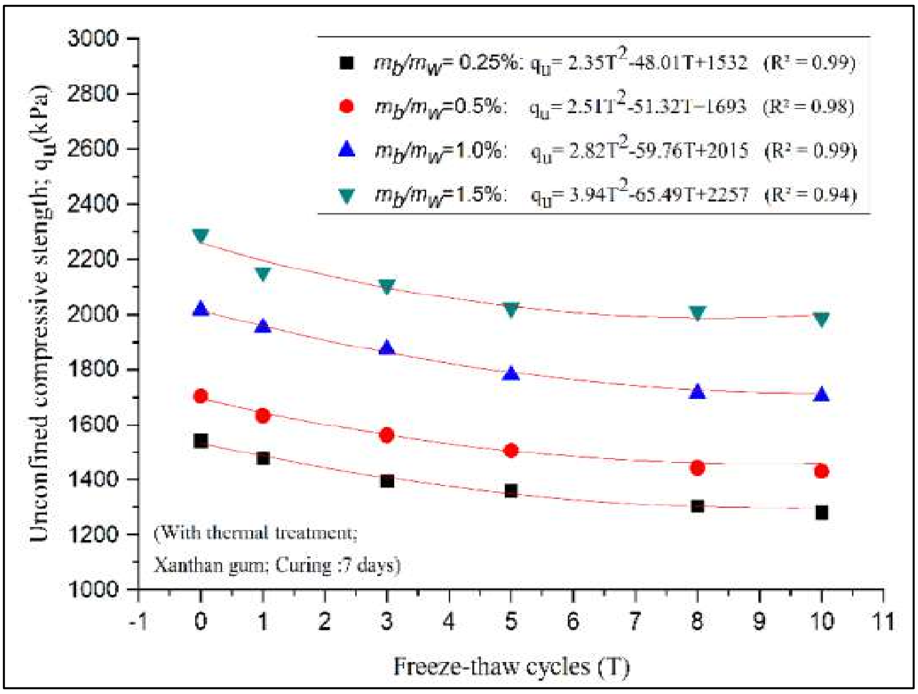
(a)


m_b/m_w=0.25%: q_u= 2.91T^2-70.55T+1604.8 (R^2 = 0.98)
m_b/m_w=0.5%: q_u= 1.82T^2-60.16T−1967.2 (R^2 = 0.99)
m_b/m_w=1.0%: q_u= 3.10T^2-74.69T+2250.6 (R^2 = 0.97)
m_b/m_w=1.5%: q_u= 2.89T^2-75.03T+2461.2 (R^2 = 0.97)
Unconfined compressive stength; q_u(kPa)
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
(With thermal treatment;
Guar gum; Curing :14 days)
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)

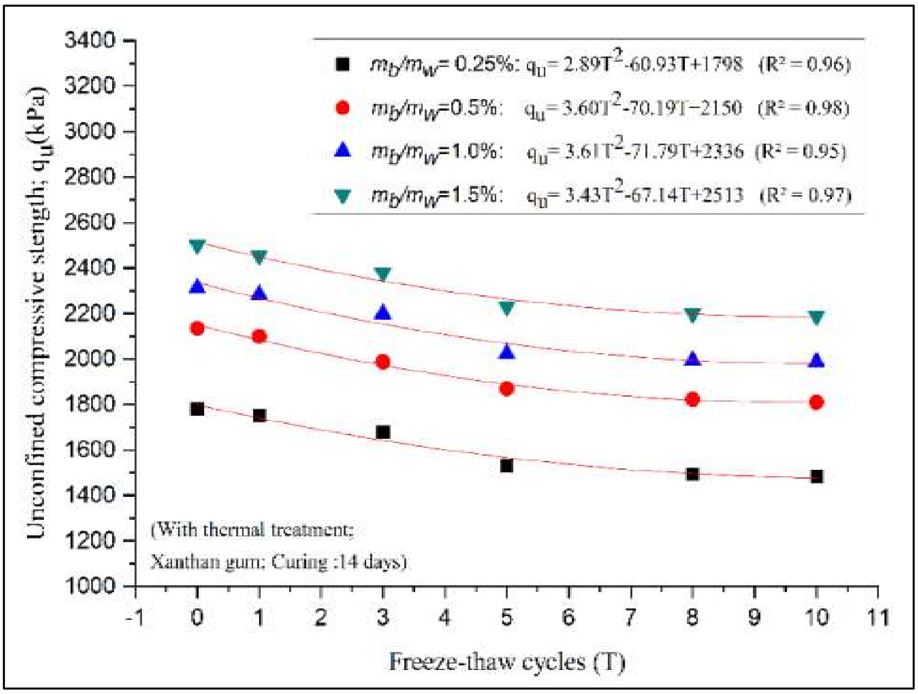
(b)


m_b/m_w=0.25%: q_u= 2.39T^2-63.70T+1951.9 (R^2 = 0.96)
m_b/m_w=0.5%: q_u= 4.64T^2-92.07T−2333.2 (R^2 = 0.95)
m_b/m_w=1.0%: q_u= 2.67T^2-73.25T+2586.9 (R^2 = 0.98)
m_b/m_w=1.5%: q_u= 4.04T^2-86.00T+2728.7 (R^2 = 0.99)
Unconfined compressive stength; q_u(kPa)
3600
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
(With thermal treatment;
Guar gum; Curing :28 days)
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)

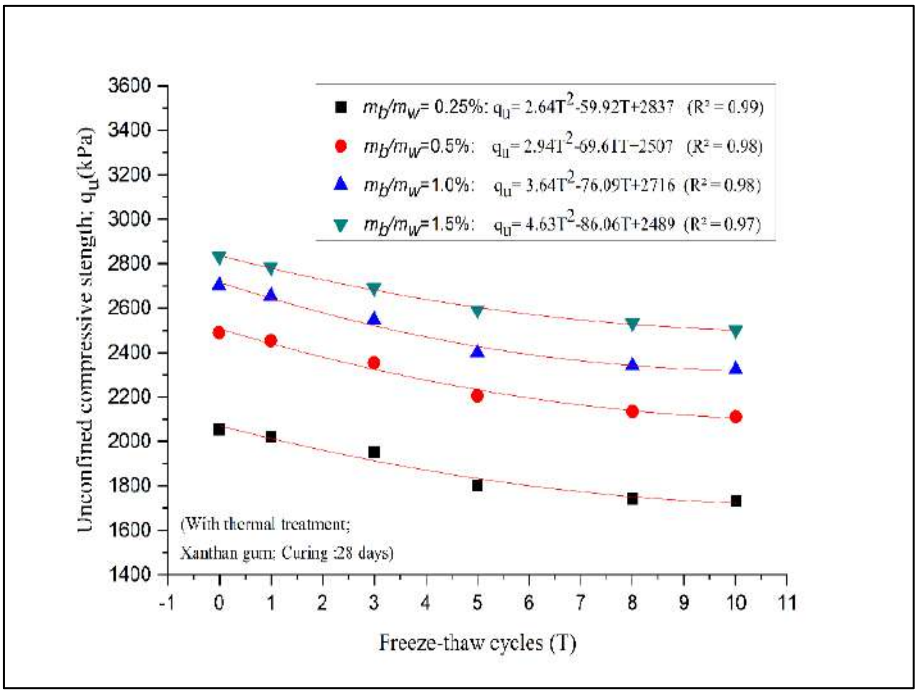
(c)


Unconfined compressive stength; q_u(kPa)
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)
m_b/m_w= 0.25%: q_u= 2.35T^2-48.01T+1532 (R² = 0.99)
m_b/m_w=0.5%: q_u= 2.51T^2-51.32T−1693 (R² = 0.98)
m_b/m_w=1.0%: q_u= 2.82T^2-59.76T+2015 (R² = 0.99)
m_b/m_w=1.5%: q_u= 3.94T^2-65.49T+2257 (R² = 0.94)
(With thermal treatment;
Xanthan gum; Curing :7 days)

(d)


Unconfined compressive stength; q_u(kPa)
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
-1
0
1
2
3
4
5
6
7
8
9
10
11
Freeze-thaw cycles (T)
m_b/m_w= 0.25%: q_u= 2.89T^2-60.93T+1798 (R² = 0.96)
m_b/m_w=0.5%: q_u= 3.60T^2-70.19T−2150 (R² = 0.98)
m_b/m_w=1.0%: q_u= 3.61T^2-71.79T+2336 (R² = 0.95)
m_b/m_w=1.5%: q_u= 3.43T^2-67.14T+2513 (R² = 0.97)
(With thermal treatment;
Xanthan gum; Curing :14 days)

(e)



(f)

Figure 3.27 Effect of F-T cycles on stabilized bauxite residue with thermal treatment at varying biopolymer concentration and curing time: (a) GG(7D), (b) GG(14D), (c) GG(28D), (d) XG(7D), (e) XG(14D), and (f) XG(28D)

3.5.5 Behavior of stabilized bauxite residue samples Post F-T cycles

This study has been performed to understand the behavior of biopolymer stabilized bauxite residue after exposure to the F-T cycles. The basic concept is to simulate the condition of any engineered structure when the temperature remains consistently above zero after a period of the wide variation in temperature (-20 to 25 ± 5 °C) during exposure to F-T cycles. The influence of F-T cycles on the durability of bauxite residue samples cannot be ignored during the construction of an engineered structure. Therefore, a series of UCS tests have been performed to determine the strength after 10 F-T cycles plus 7, 14, and 28 days of post F-T cycles curing at the same conditions used for 28 days of curing time before exposure to F-T cycles.

The influence of F-T cycles on the mechanical characteristics of biopolymer stabilized RMT resulted in a decrease of unconfined compressive strength to the tune of 23.3, 16.4, 19.7, and 11.6% for GG(WTT), GG(TT), XG(WTT), and XG(TT) respectively. The recovery in compressive strength was evaluated from the test for up to 75 days (Figure 3.28) and showed that it almost remained constant after 14 days of curing for all the biopolymer (XG and GG) stabilized samples with and without thermal treatment. The recovery of unconfined compressive strength, Post F-T cycles, cured for 7, 14, and 28 days was recorded as 16.4, 11.5, 12.4, and 8.5% for GG(WTT), GG(TT), XG(WTT), and XG(TT) respectively. The recovery in strength is possibly due to the biopolymer efficacy of binding the red mud particles sustained even after exposure to F-T cycles.

The loss of strength after 10 F-T cycles and recovery of strength post F-T cycles conditioning can be represented mathematically as shown in equations (1) and (2) and graphically as shown in Figure 3.29.

UCS (kPa) at 28 days = $q_{u\ (28)}$

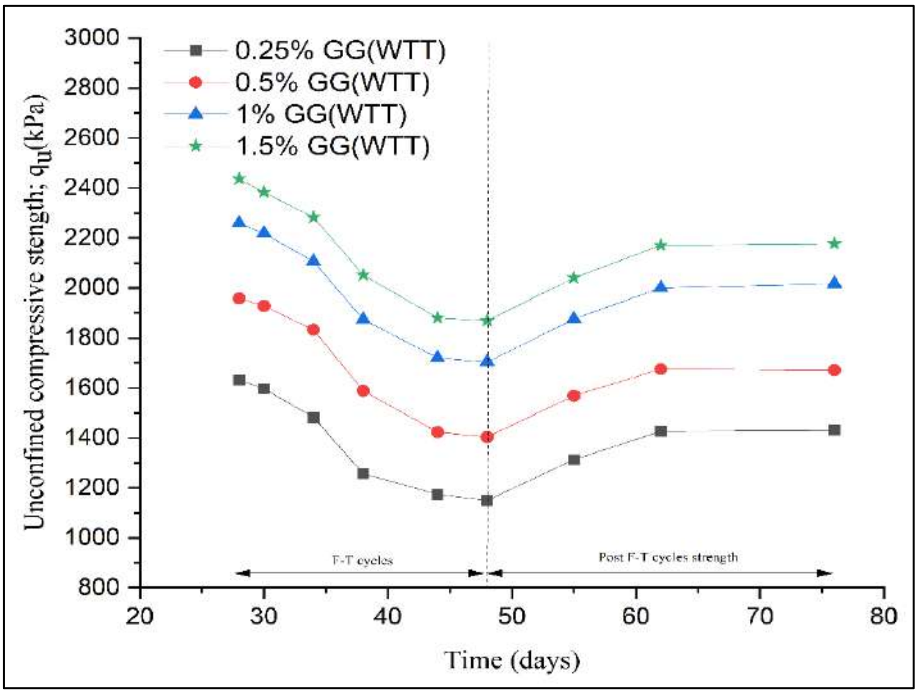
UCS (kPa) after 10 F-T cycles = $q_{u\ (10\ \text{F-T cycles})}$

UCS (kPa) post 10 F-T cycles conditioning = $q_{u\ (\text{Post } 10\ \text{F-T cycles})}$

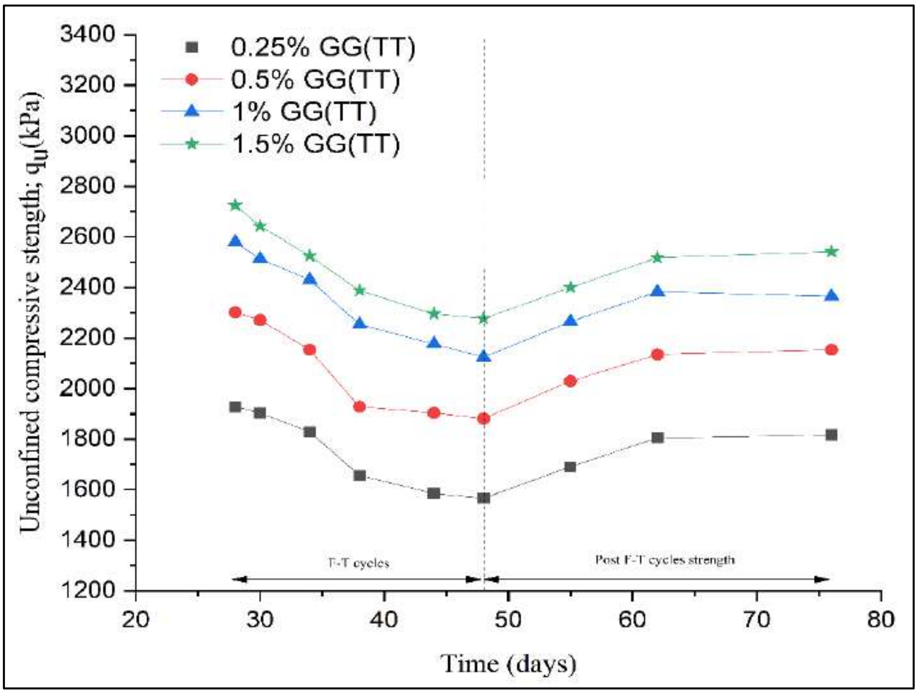
Loss of strength = $q_{u\ (28)} - q_{u\ (10\ \text{F-T cycles})} / q_{u\ (28)}$ .... (1)

Recovery of strength = $q_{u\ (\text{Post } 10\ \text{F-T cycles})} - q_{u\ (10\ \text{F-T cycles})} / q_{u\ (10\ \text{F-T cycles})}$ .... (2)

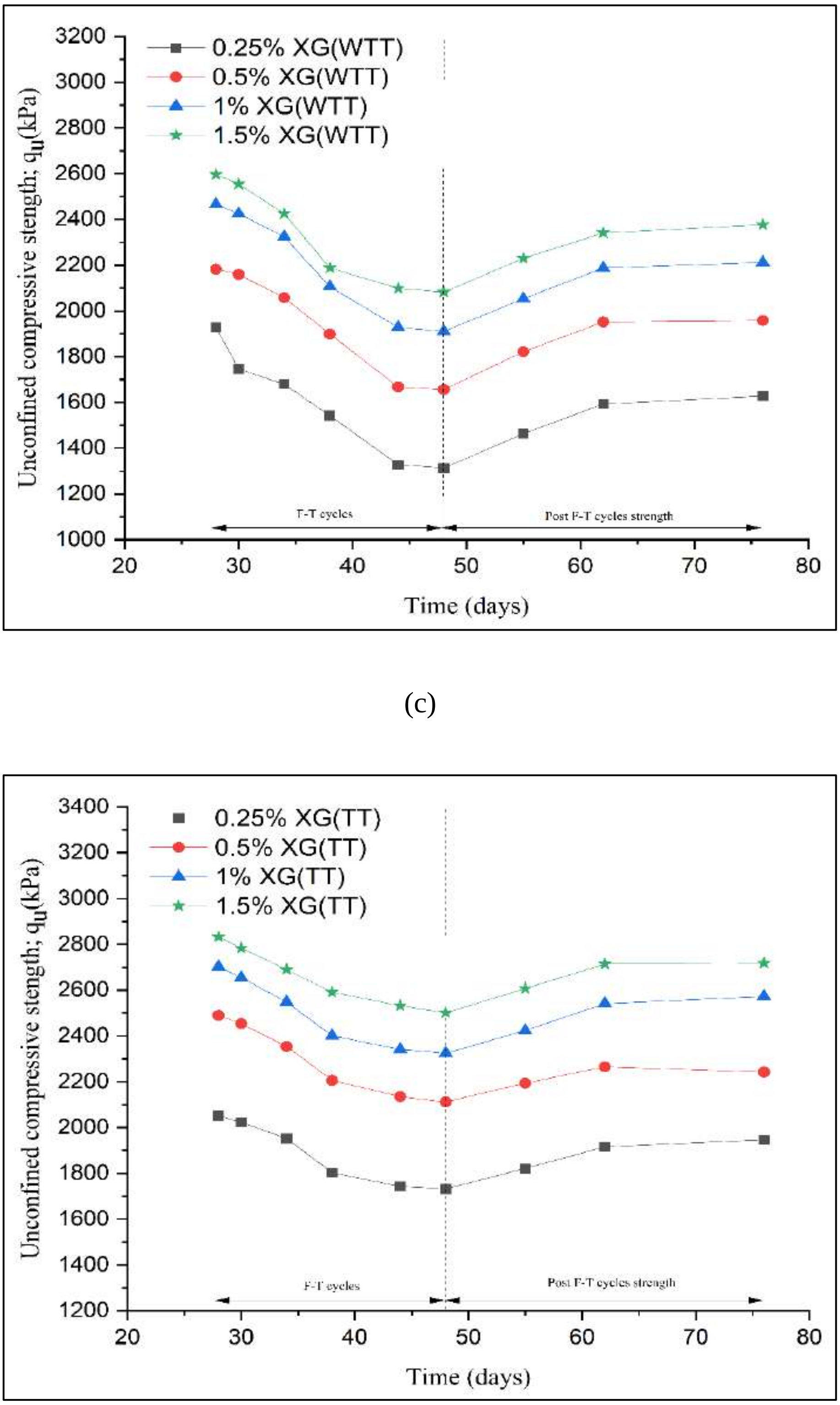
As it is evident that there is a substantial gain in compressive strength post F-T cycles, if allowed to cure further, the suggestion to avoid construction problems is to stop the work for a minimum of 48-72 h until the temperature remain consistently above 0 ºC so that enough recovery of compressive strength is achieved.


3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
Unconfined compressive stength; q_u(kPa)
0.25% GG(WTT)
0.5% GG(WTT)
1% GG(WTT)
1.5% GG(WTT)
F-T cycles
Post F-T cycles strength
20
30
40
50
60
70
80
Time (days)

(a)


3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
Unconfined compressive stength; q_u(kPa)
0.25% GG(TT)
0.5% GG(TT)
1% GG(TT)
1.5% GG(TT)
F-T cycles
Post F-T cycles strength
20
30
40
50
60
70
80
Time (days)

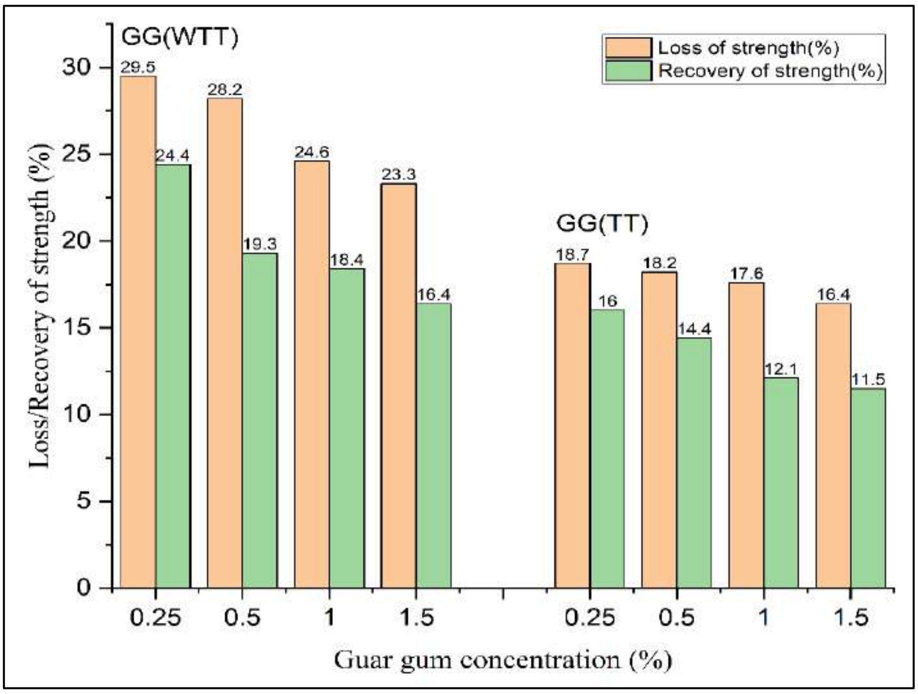
(b)



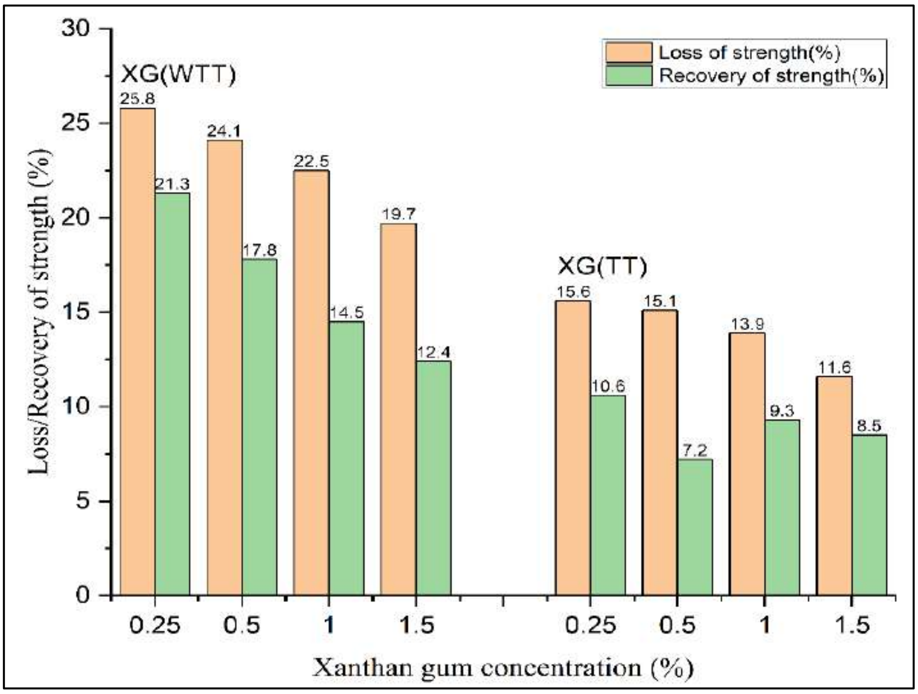
(c)

(d)

Figure 3.28 UCS (q_u) versus time during F-T process and post F-T conditioning: (a) GG(WTT), (b) GG(TT), (c)XG(WTT), and (d) XG(TT)



(a)



(b)

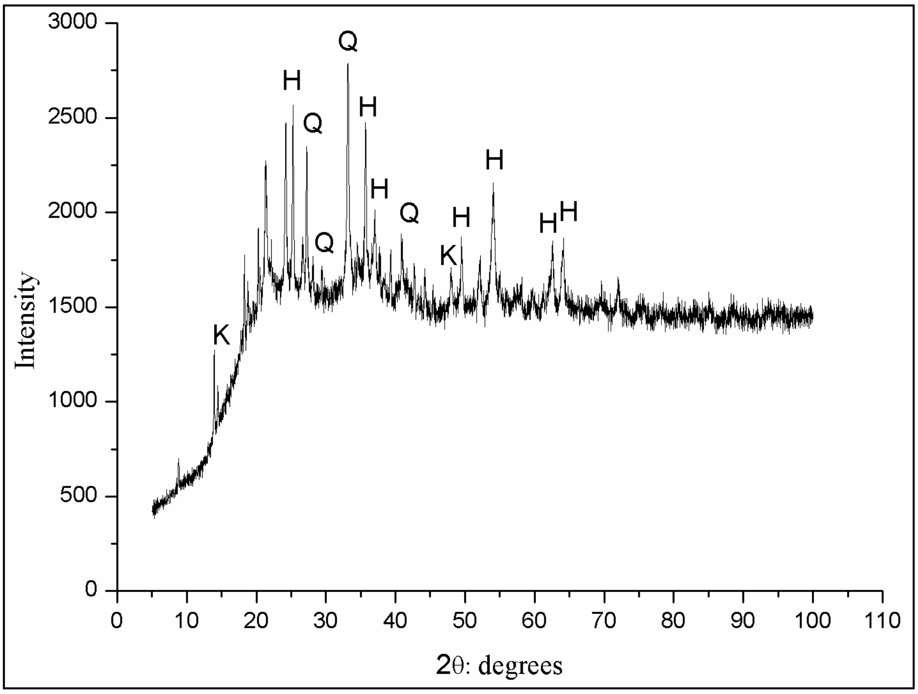
Figure 3.29 Loss of strength (after 10 F-T cycles) and Recovery of strength (post F-T cycles conditioning)

3.6 Mineralogical and Morphological Study

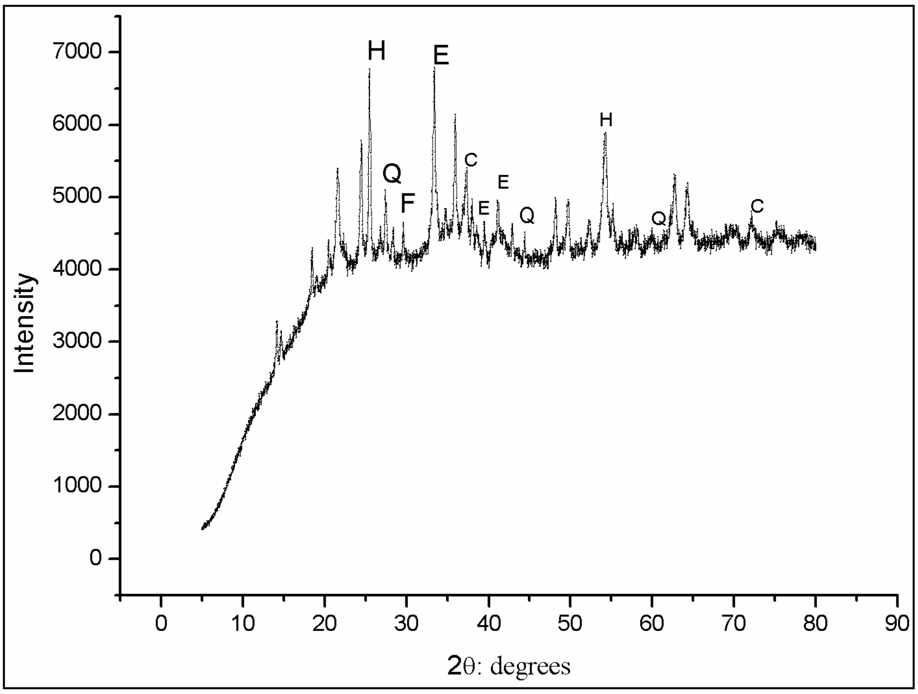
The bauxite residue samples from the strength tests with different concentration of biopolymer and curing periods were examined for their mineralogical changes using X-ray diffraction (XRD) test and their morphological changes by utilizing scanning electron microscope (SEM).

3.6.1 Mineralogical study (X-Ray diffraction)

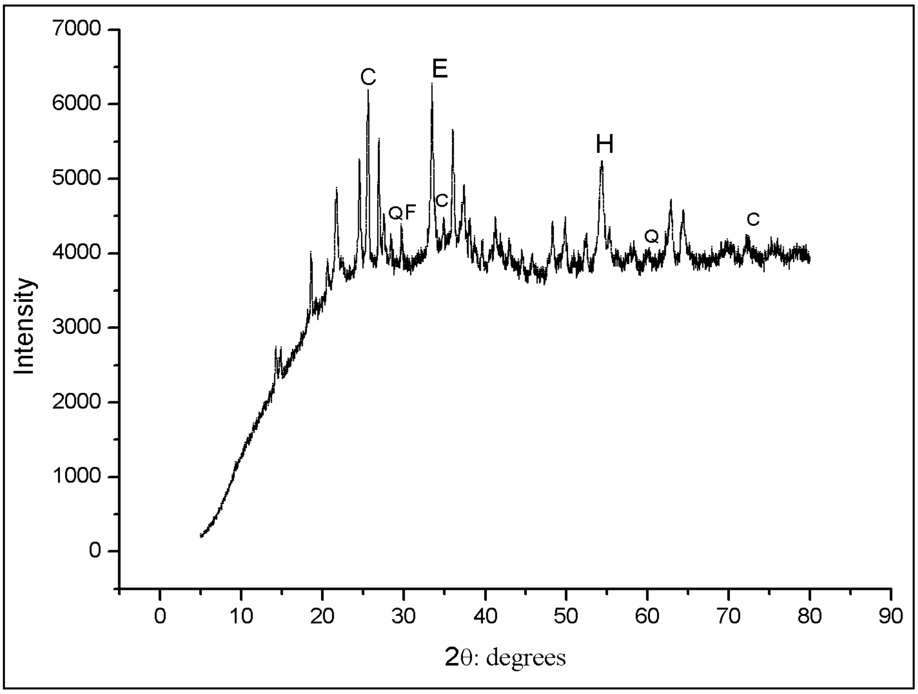
As discussed in the section 2.3.8, the X-ray diffraction (XRD) on bauxite residue samples was performed using the MiniFlex X-ray diffractometer from Rigaku. The observation is based on identifying the presence of unknown crystalline materials (e.g., minerals, inorganic compounds). The XRD patterns of untreated bauxite residue and bauxite residue stabilized with 1.5% guar gum and 1.5% xanthan gum are shown in Figure 3.30. The significant minerals observed are Quartz (Q), Hematite (H), Eskolaite (E), Corundum (C), Kaolinite (K), and Fluorite (F).



(a)



(b)



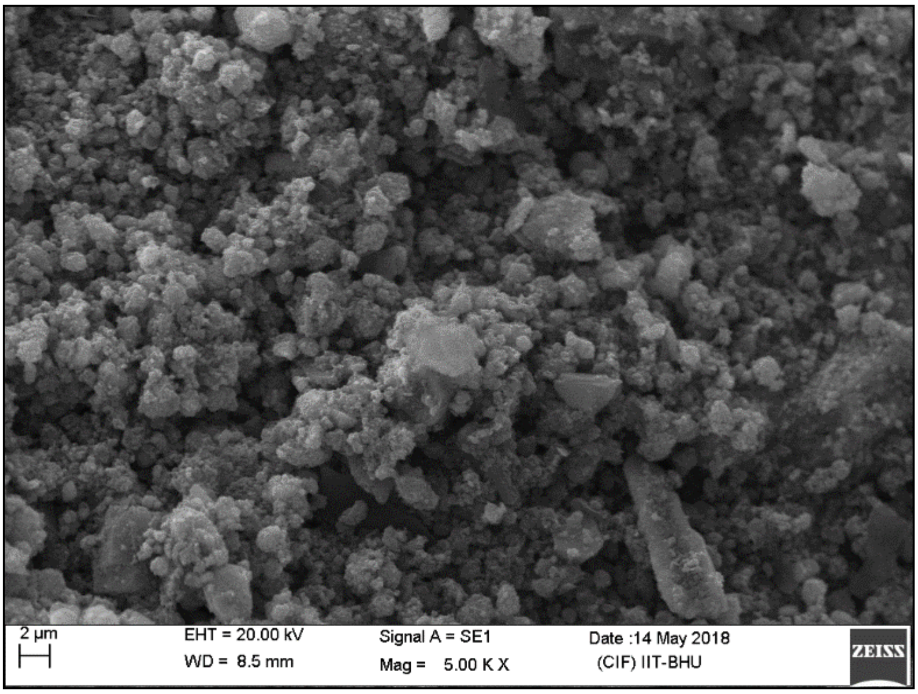
(c)

Figure 3.30 XRD analysis of untreated and biopolymer stabilized bauxite residue sample at 1.5% concentration: (a) Untreated bauxite residue, (b) guar gum, and (c) xanthan gum.

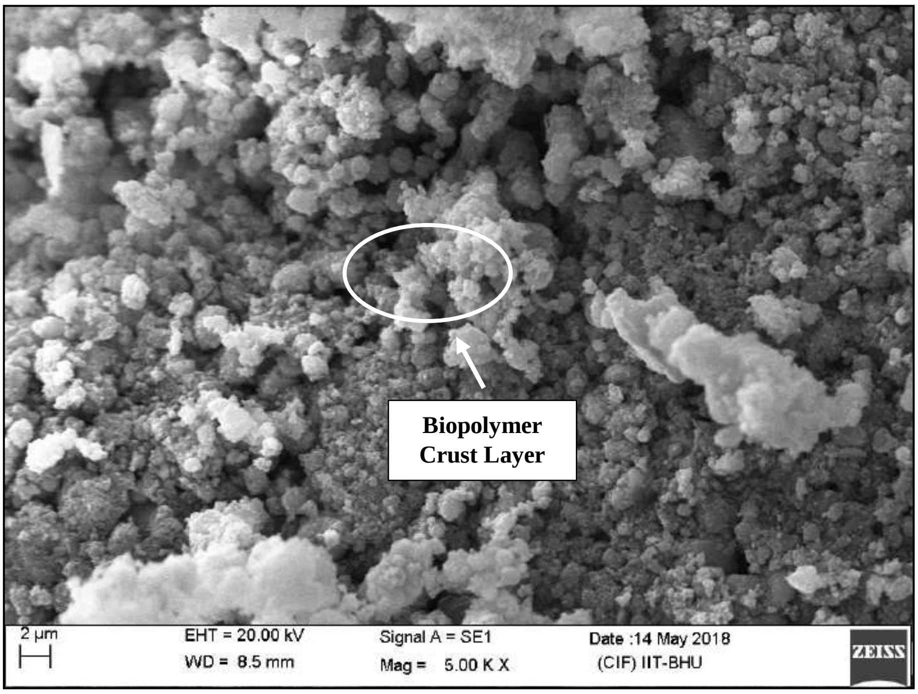
All the results show good narrow peeks showing crystallinity and excellent regularity in particular directions. Crystallinity is a measure of uniformity in the arrangement of structural elements. The crystallinity has been measured on the evidence that an increase in amorphousness tends to broaden the line width; whereas, an increase in crystallinity increases the intensity. An increase in the crystallinity of the biopolymer-bauxite residue mix also attributes to its higher strength [95]. The stabilized bauxite residue also shows higher peak intensity than the untreated bauxite residue sample evidencing biopolymer effectiveness in enhancing the strength.

3.6.2 Morphological study (Scanning Electron Microscopy)

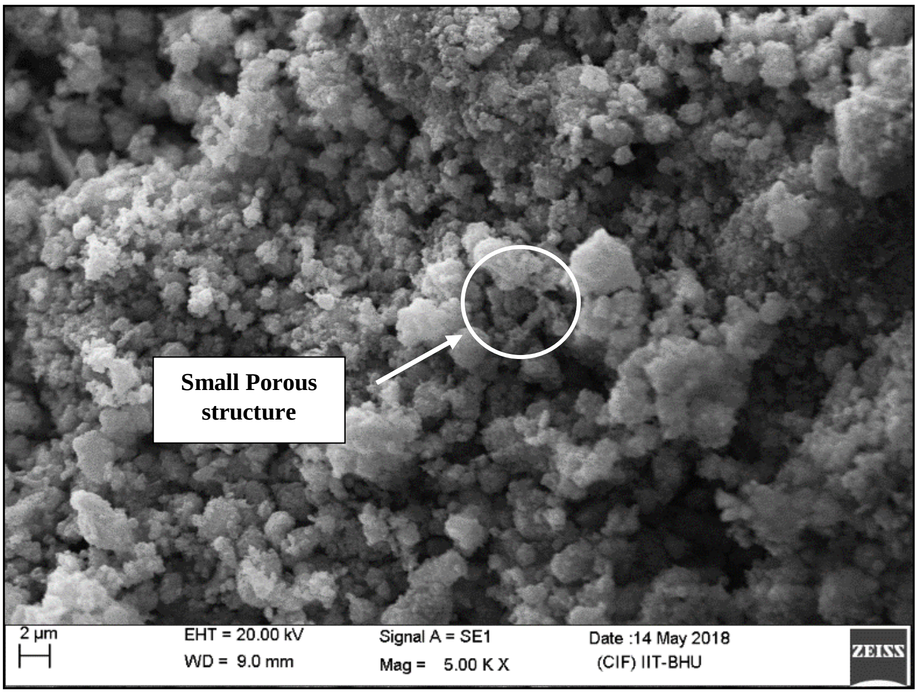
The morphological changes of the untreated bauxite residue and biopolymer stabilized bauxite residue samples at 1.5% concentration are shown in Figure 3.31. The imaging was performed using a ZEISS EVO 18 SEM from ZEISS Microscopy at a magnification of 5.00 kX. The SEM images revealed the formation of biopolymer aggregates on bauxite residue samples that increased with increasing concentration. The untreated bauxite residue sample with only water added exhibits a loose structure with voids; whereas, biopolymer treated bauxite residue samples had a denser structure with particles bonded and voids clogged with biopolymer gel coating. Also, in Figure 3.31(e), the SEM image shows that bauxite residue, thermally treated with 1.5% xanthan gum and exposed to 10 F-T cycles, exhibit small voids and cavities but sufficient strength compared to untreated bauxite residue. Chen [96] also observed similar behavior.


2 µm
EHT = 20.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 5.00 K X
Date :14 May 2018
(CIF) IIT-BHU
ZEISS

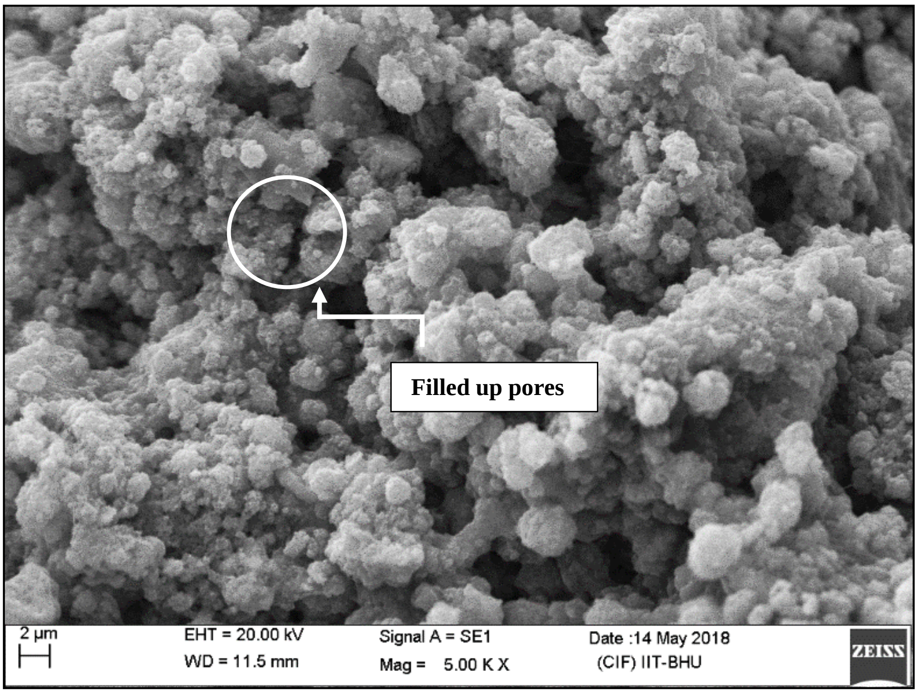
(a)


Biopolymer Crust Layer
2 µm
EHT = 20.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 5.00 K X
Date :14 May 2018
(CIF) IIT-BHU
ZEISS

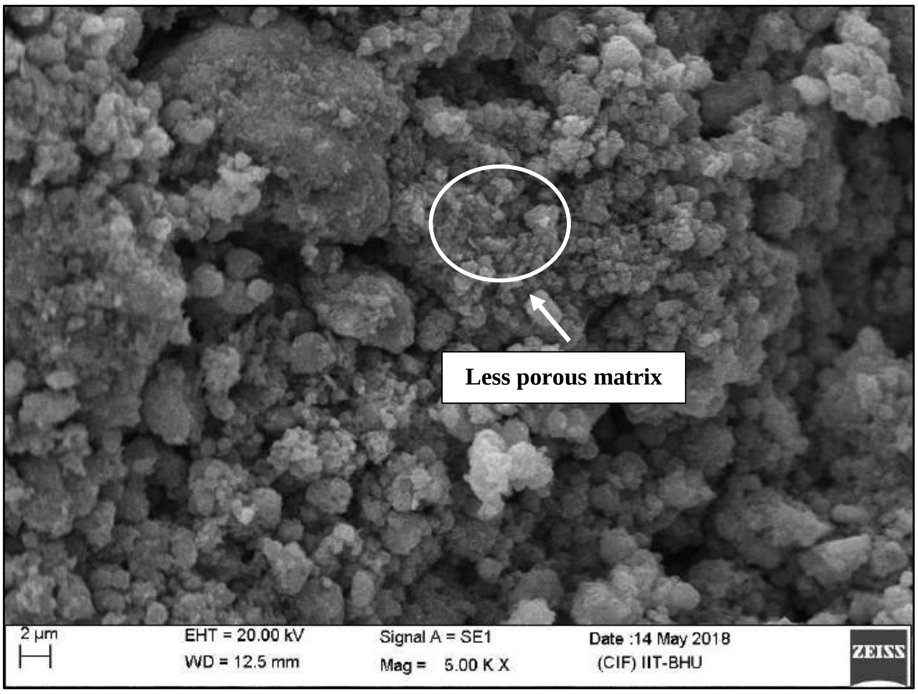
(b)


Small Porous structure
2 µm
EHT = 20.00 kV
WD = 9.0 mm
Signal A = SE1
Mag = 5.00 K X
Date :14 May 2018
(CIF) IIT-BHU
ZEISS

(c)


Filled up pores
2 µm
EHT = 20.00 kV
WD = 11.5 mm
Signal A = SE1
Mag = 5.00 K X
Date :14 May 2018
(CIF) IIT-BHU
ZEISS

(d)



(e)

Figure 3.31 SEM micrographs of bauxite residue samples: (a) untreated, (b) 1.5% GG, (c) 1.5% XG, (d) 1.5% (XG+GG), and (e) 1.5% XG, 28 days curing time and 10 F-T cycles