CHAPTER - 3

RESULTS AND DISCUSSION

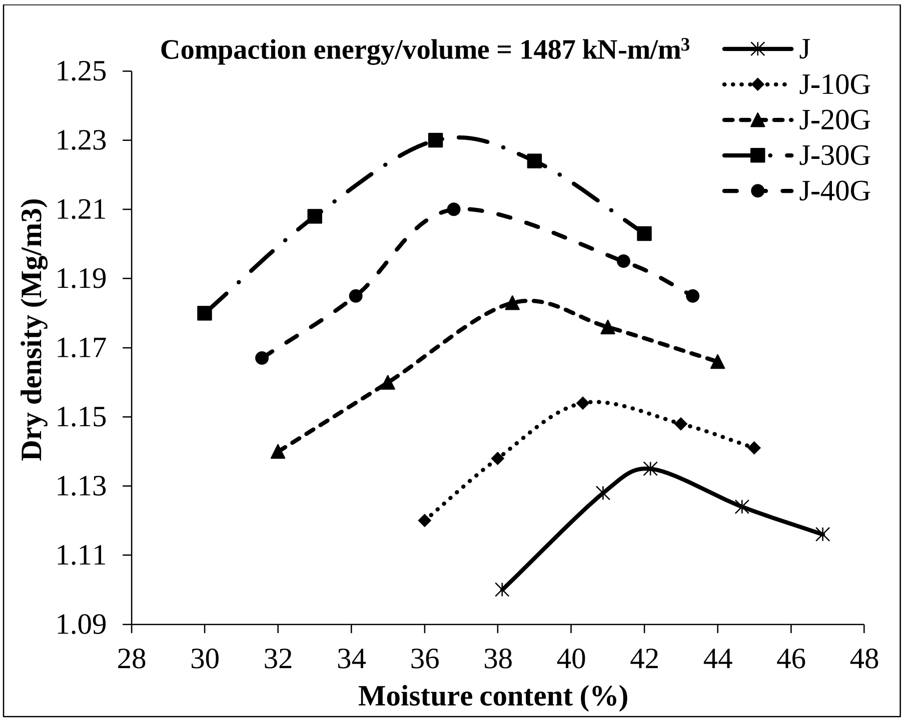
This chapter describes the experimental studies conducted in laboratory along with their results and discussions. The experimental studies includes, Compaction (Mini compaction mold), Strength (Unconfined compressive and Split tensile strength tests), Durability (Freezing-Thawing study), Mineralogical and Morphological (XRD & SEM-EDX tests) and Leachate (TCLP-ICP) have been carried out on jarosite waste stabilized with GGBS and lime.

3.1 Compaction Study

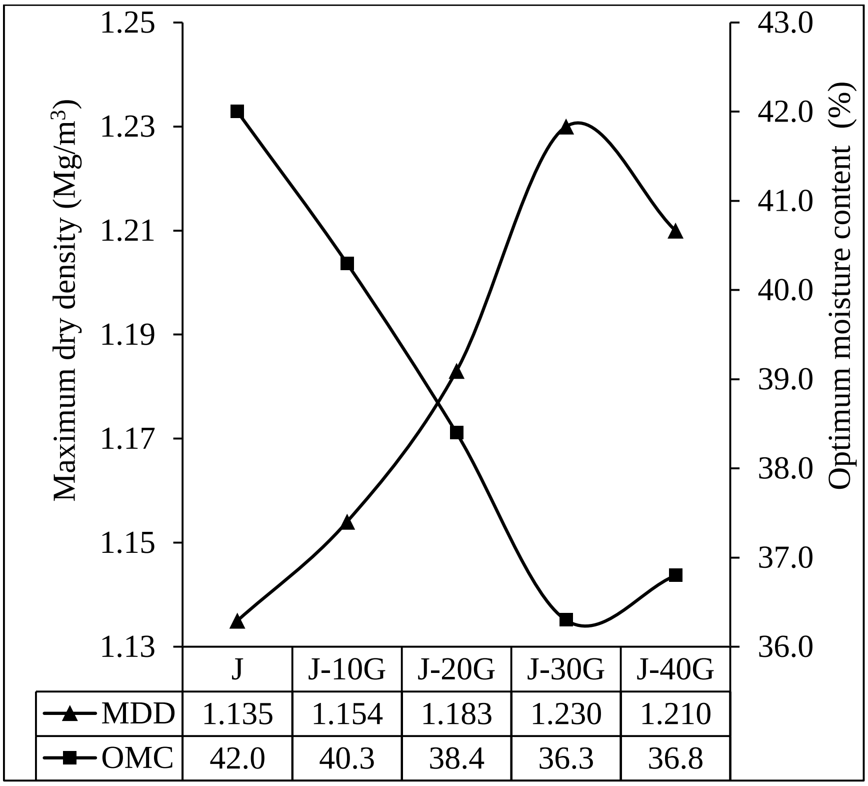
In the methodology section discussed in Chapter 2, The Mini Compaction Mould, advanced by Sridharan and Sivapullaiah [60], was used for evaluation of compaction parameters such as Maximum dry density (MDD) and Optimum moisture content (OMC).

3.1.1 Effect of GGBS on compaction parameters

The variations in the moisture content and dry density of jarosite blended with varying percentage of ground granulated blast furnace slag (GGBS) (10, 20, 30 and 40% by weight of dry jarosite) are shown in Figure 3.1 (a). It was observed that the MDD and OMC of the jarosite were 1.13 Mg/m^3 and 42% respectively, which changed after blending with GGBS and a decrease in the OMC and an increase in the MDD were observed with up to 30% GGBS. With further increase in GGBS content (40%), an increase in the OMC and a decrease in the MDD were observed. The variations in the MDD and OMC of jarosite blended with varying percentage of GGBS are shown in Figure 3.1 (b).



(a)



	J	J-10G	J-20G	J-30G	J-40G
MDD	1.135	1.154	1.183	1.230	1.210
OMC	42.0	40.3	38.4	36.3	36.8

(b)

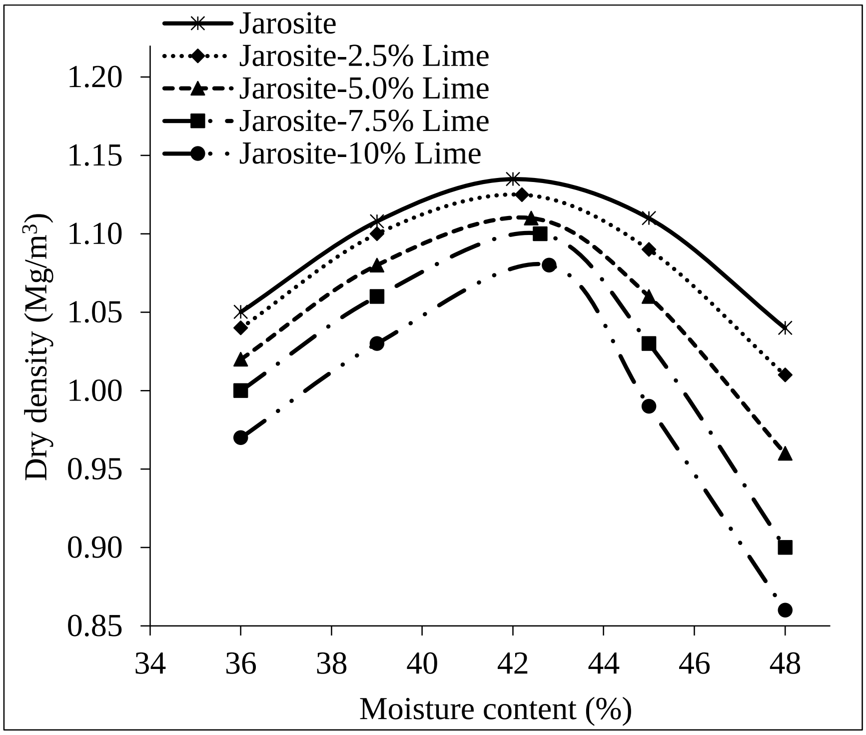
Figure 3.1 Compaction characteristics of jarosite with GGBS (a) Effect of GGBS on dry density and moisture content; (b) Variation of MDD and OMC with GGBS

This trend in variation of MDD and OMC for various jarosite-GGBS mixtures is mainly due to the modification of the arrangement of particles in the blend. Primarily, up to 30% GGBS, void spaces among the jarosite particles are occupied by the GGBS particles and the matrix changes from a flocculated to dispersed structure, and hence, the MDD increases with an increase in GGBS content. Afterward, the additional amount of GGBS added leads to the separation of particles in the matrix causing the reduction in MDD.

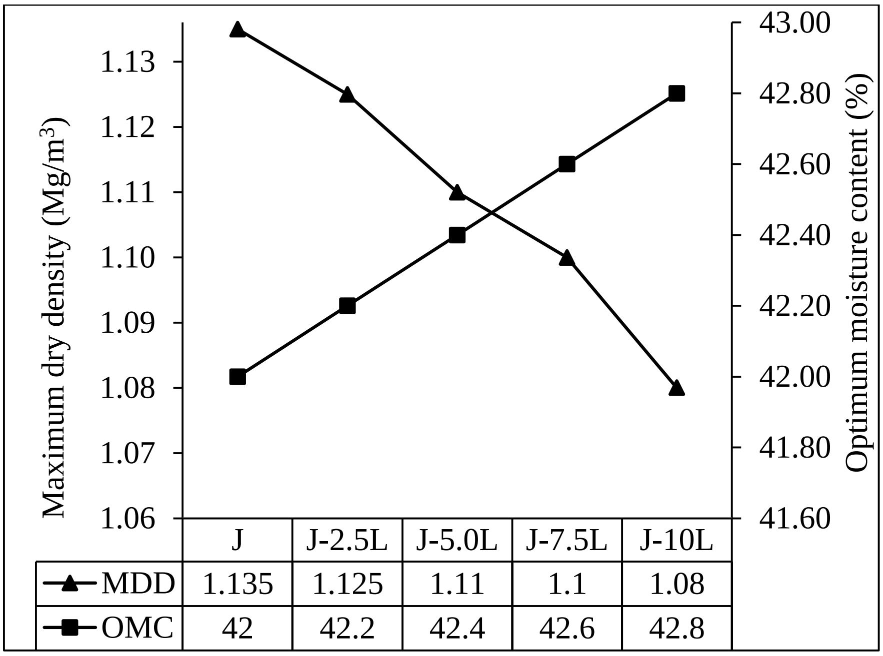
Also, addition of GGBS causes decrease in OMC of the jarosite-GGBS mixture because of the less specific surface area present in GGBS as compared to jarosite and its glassy nature, affecting its interaction with moisture differently than the jarosite. Thus, requires a lower amount of moisture content to attain MDD. Further, with the addition of 40% GGBS, segregation of particles happened leading to an increase in the OMC.

3.1.2 Effect of lime and GGBS on compaction parameter

All the jarosite-GGBS mixtures with different GGBS content were further blended with varying percentage of hydrated lime i.e., 2.5, 5.0, 7.5 and 10%. The minimum lime percentage required for jarosite stabilization was 2.5% and was determined by using Eades and Grim test method (ASTM C977-18 [82a]), whereas, 5, 7.5 and 10% of lime content were also used, based on the international experiences with soil-lime stabilization [82b]. The variations in the OMC and MDD for various blends of jarosite, lime and GGBS are shown in Figures 3.2 to 3.5 and it is observed that after addition of lime in GGBS amended jarosite, an increase in the OMC and a decrease in the MDD was observed. This happened due to the lower specific gravity of lime compared with jarosite and GGBS, hence, higher moisture content is required to lubricate the particles of the composite mix to attain its MDD and OMC. Furthermore, the higher percentage of moisture content helped in acceleration of the pozzolanic reactions (more agglomeration formed); thus, adding of hydrated lime (active calcium) with GGBS (active siliceous) enhanced the compactness of treated jarosite and hence it is advantageous to use GGBS and lime in the jarosite stabilization. Table 3.1 shows the summary of compaction parameters influenced by the addition by GGBS and lime as a binder in jarosite.

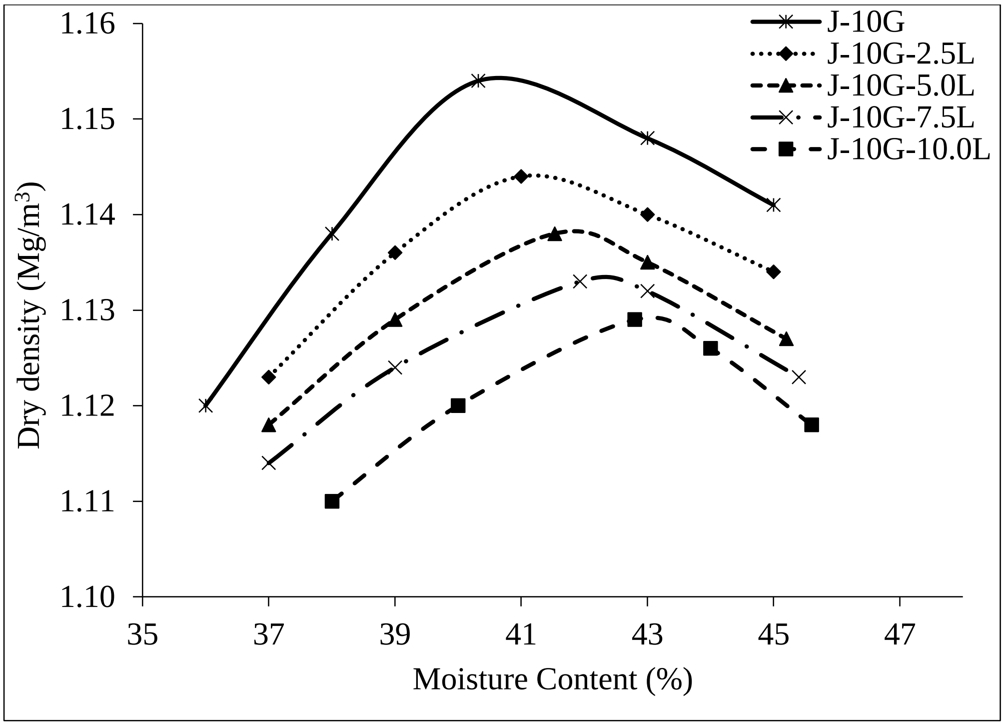


(a)

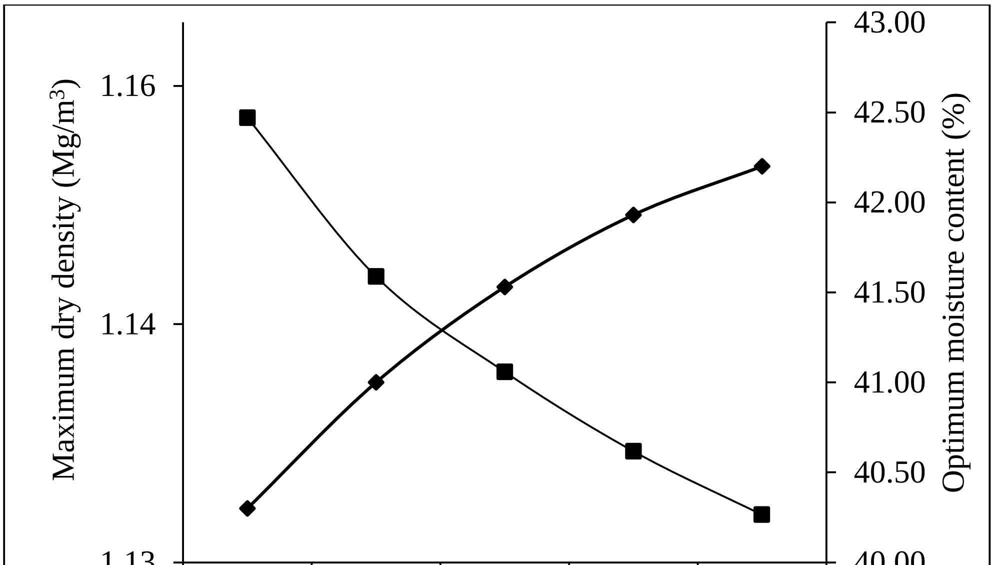


(b)

Figure 3.2 Compaction characteristics of jarosite-lime blends (0% GGBS) (a) Effect of lime on dry density and moisture content; (b) Variation of MDD and OMC with lime



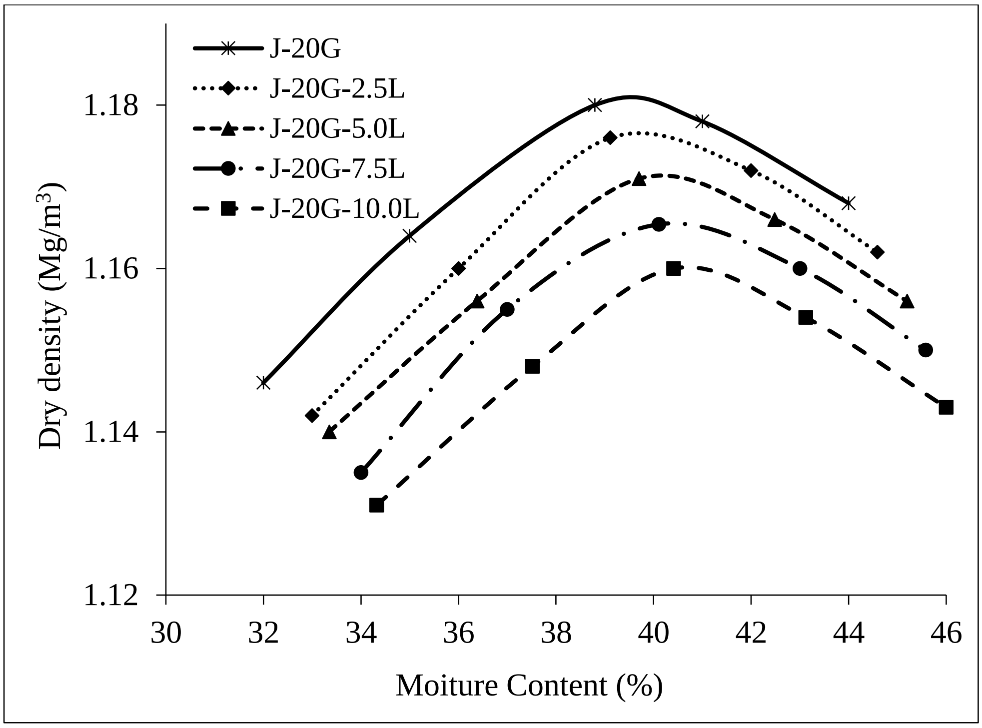
(a)



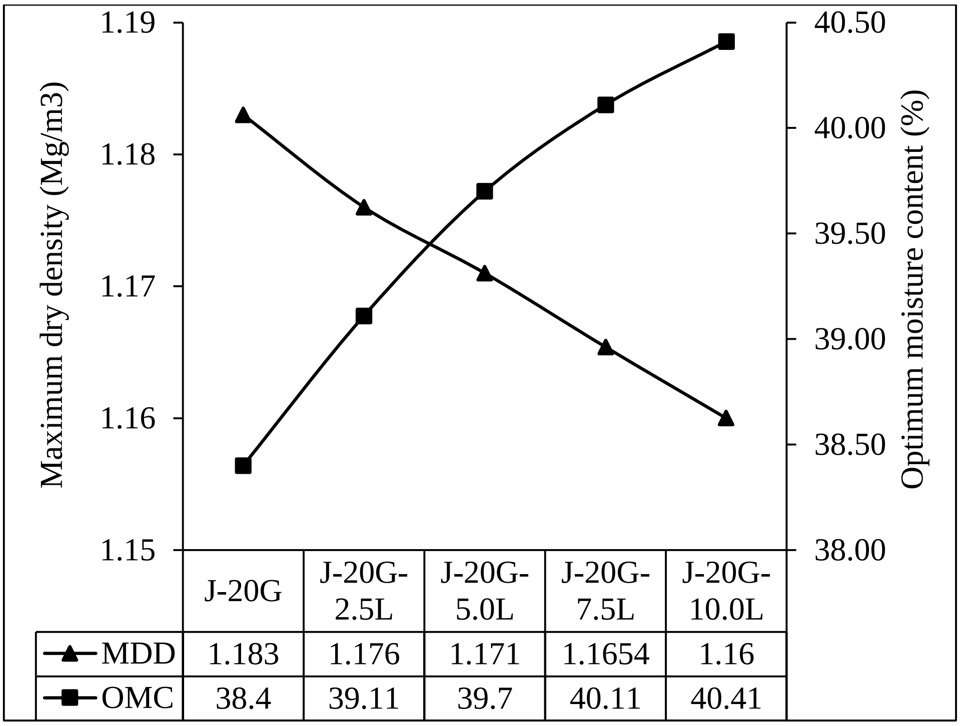
	J-10G	J-10G-2.5L	J-10G-5.0L	J-10G-7.5L	J-10G-10.0L
MDD	1.154	1.144	1.138	1.133	1.129
OMC	40.3	41	41.53	41.93	42.2

(b)

Figure 3.3 Compaction characteristics of jarosite-10% GGBS blend with lime (a) Effect of lime on dry density and moisture content; (b) Variation of MDD and OMC with lime



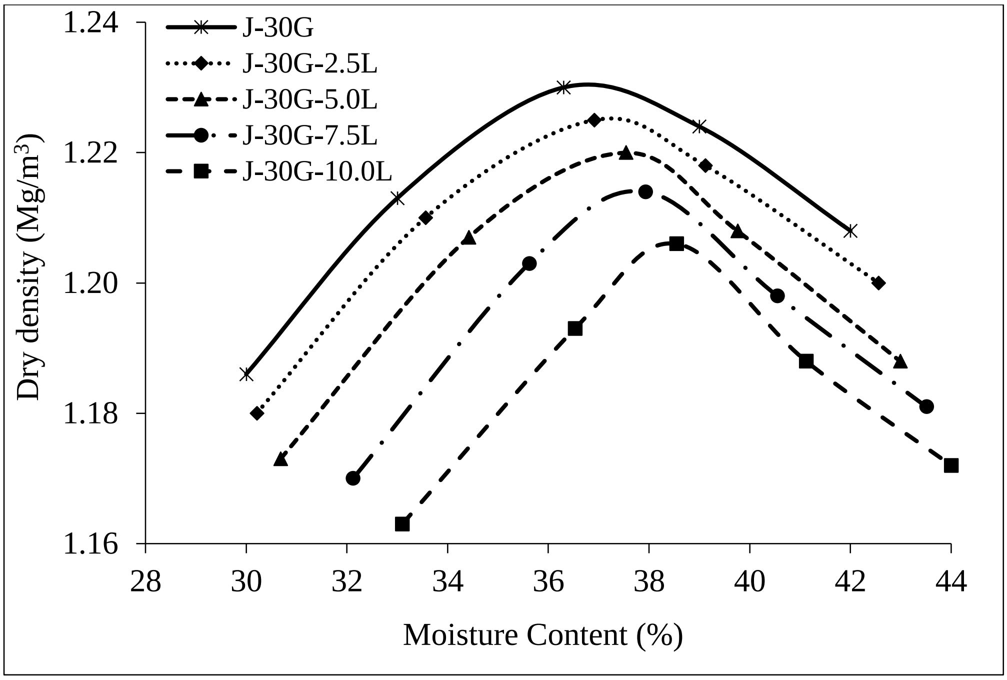
(a)



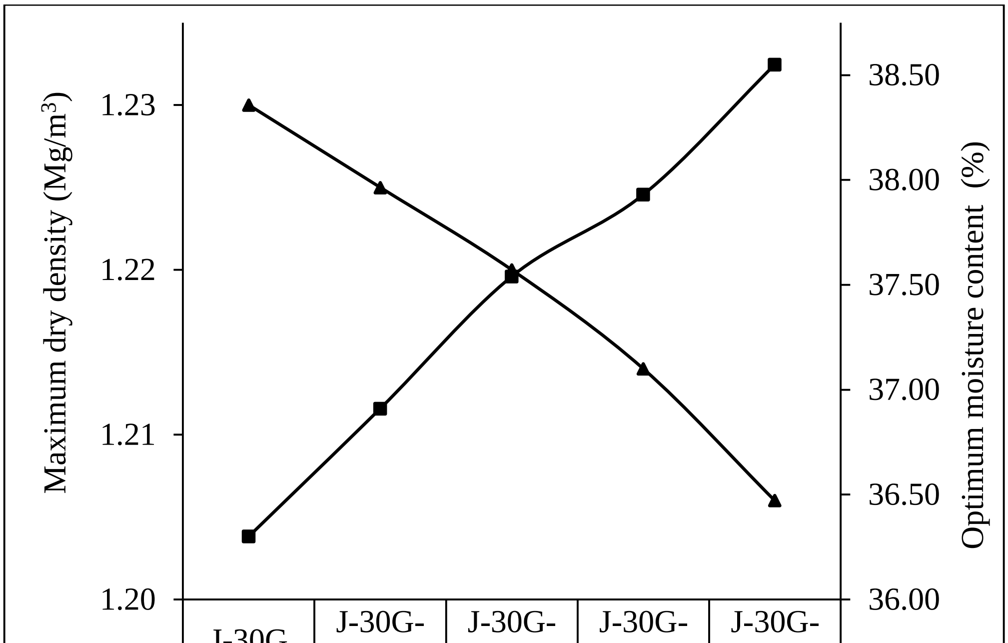
	J-20G	J-20G-2.5L	J-20G-5.0L	J-20G-7.5L	J-20G-10.0L
MDD	1.183	1.176	1.171	1.1654	1.16
OMC	38.4	39.11	39.7	40.11	40.41

(b)

Figure 3.4 Compaction characteristics of jarosite 20% GGBS with lime (a) Effect of lime on dry density and moisture content; (b) Variation of MDD and OMC with lime



(a)



	J-30G	J-30G-2.5L	J-30G-5.0L	J-30G-7.5L	J-30G-10.0L
MDD	1.230	1.225	1.22	1.214	1.206
OMC	36.3	36.91	37.54	37.93	38.55

(b)

Figure 3.5 Compaction characteristics of jarosite-30% GGBS with lime (a) Effect of lime on dry density and moisture content; (b) Variation of MDD and OMC with lime

Table 3.1 Summary of compaction parameters of the various jarosite-GGBS-lime mixtures

Mixtures	Proportions	MDD (Mg/m^3)	OMC (%)
Jarosite	Untreated	1.135	42.09
Jarosite-Lime	J+2.5% L	1.125	42.23
	J+5.0% L	1.110	42.47
	J+7.5% L	1.100	42.63
	J+10% L	1.080	42.89
Jarosite-GGBS	J+10% G	1.154	40.32
Jarosite-GGBS-Lime	J+10% G+2.5% L	1.144	41.09
	J+10% G+5.0% L	1.138	41.53
	J+10% G+7.5% L	1.133	41.96
	J+10% G+10% L	1.129	42.23
Jarosite-GGBS	J+20% G	1.183	38.40
Jarosite-GGBS-Lime	J+20% G+2.5% L	1.176	39.11
	J+20% G+5.0% L	1.171	39.70
	J+20% G+7.5% L	1.165	40.23
	J+20% G+10% L	1.160	40.47
Jarosite-GGBS	J+30% G	1.230	36.30
Jarosite-GGBS-Lime	J+30% G+2.5% L	1.225	36.91
	J+30% G+5.0% L	1.220	37.54
	J+30% G+7.5% L	1.214	37.93
	J+30% G+10% L	1.206	38.55

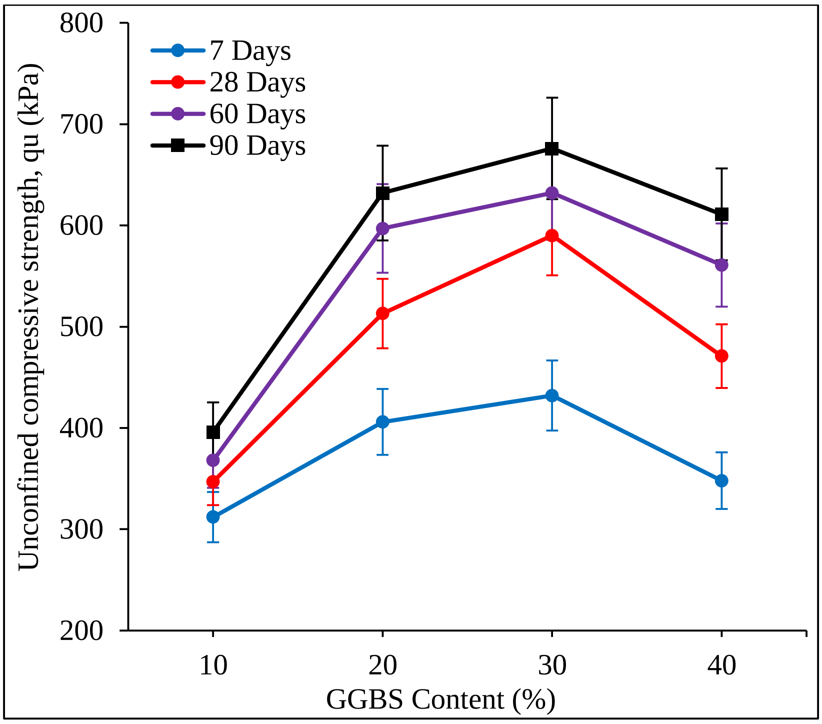
3.2 Strength Study

With the help of MDD and OMC of all the jarosite-GGBS-lime mixture, the samples for strength characteristics namely unconfined compressive strength (UCS) and split tensile strength test were prepared as per ASTM D2166-06 [62] and ASTM D-3967 [63] respectively, as discussed in the article 2.3.7.

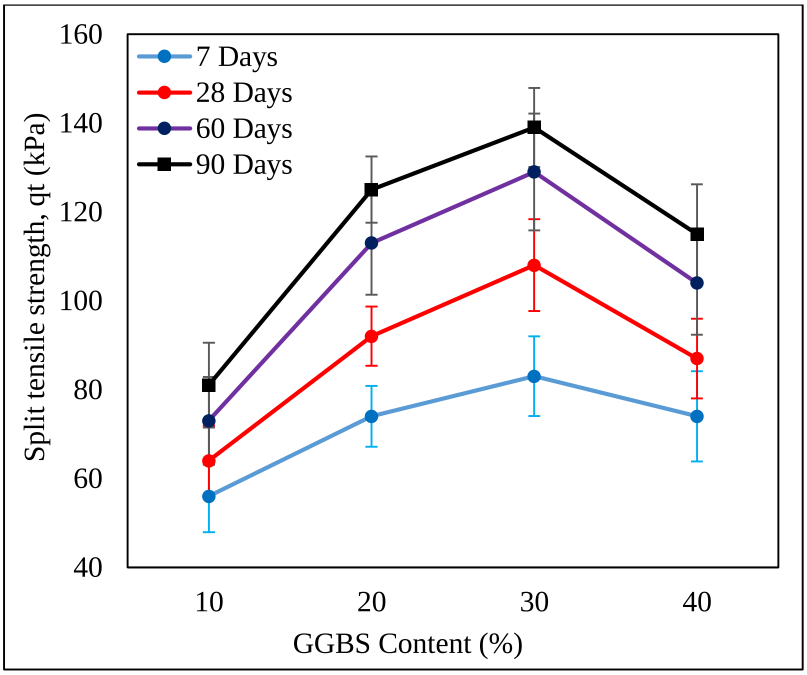
3.2.1 Effect of the GGBS on jarosite-GGBS mixture

A series of Unconfined Compressive Strength (q_u) and split tensile strength (q_t) tests were carried out on jarosite-GGBS mixtures to study the influence of GGBS on the strength characteristics for different curing period, and the outcomes are presented in Figure 3.6. It is observed from Figure 3.6 that both the strength characteristics of jarosite (UCS (q_u) and split tensile strength (q_t)) increase with the increase in GGBS content up to 30%. By further adding of GGBS, i.e. 40%, a significant reduction occurs in both, UCS (q_u) and split tensile strength (q_t). The variations observed in strength characteristics are due to increase in pozzolanic content (GGBS) and alteration in the mechanical properties of the GGBS mixtures (compaction parameters).

From Table 3.1, it is also justified that with the increase in GGBS content, the compaction parameters (MDD and OMC) are observed to vary. With up to 30% GGBS, the MDD increases and the OMC reduces as compared to jarosite alone, which is possibly the reason for the improvement in the strength characteristics because of mechanical alternation in the particles of GGBS and jarosite. However, at 40% GGBS, it is observed that the MDD decreases and the OMC increases. Hence, the strength also reduces. It is also clear from Figure 3.6 that with an increase in curing period such as 7, 28, 60 and 90 days, the strength also increases because the pozzolanic material (GGBS) has self-hardening time-dependent properties which increase with curing period. Similar findings were also reported by Sharma and Sivapullaiah [85].



(a)



(b)

Figure 3.6 Effect of GGBS content on strength of jarosite with all curing period (a) UCS (q_u) and (b) Split tensile strength (q_t)

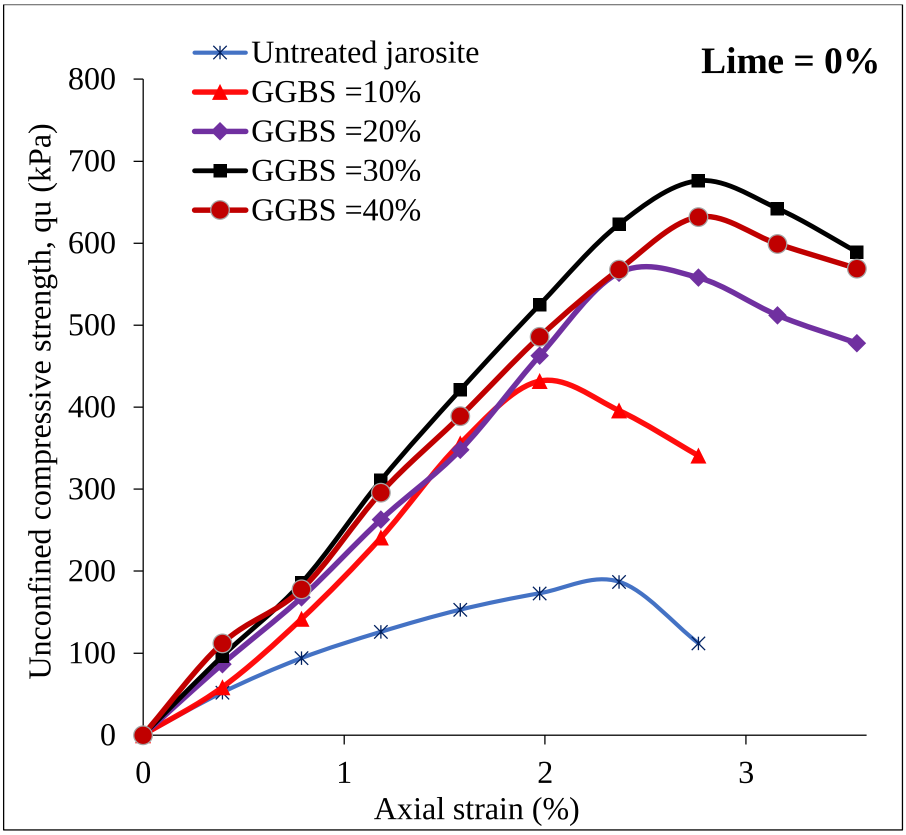
The unconfined compressive strength of jarosite-GGBS samples containing 30% GGBS increases from 187 kPa (untreated jarosite) to 473.54, 567.45, 598.23 and 622.87 kPa at 7, 28, 60 and 90 days curing period respectively. Similarly, split tensile strength of jarosite-GGBS samples containing 30% GGBS increases from 96.32 kPa (untreated jarosite) to 118.45, 136.12 and 143.28 kPa at 7, 28, 60 and 90 days curing period respectively. From both strength test results, it was noticed that higher the curing period (90 days), higher is the strength.

It is remarkable to note that the GGBS acts as a binder in the stabilization of jarosite matrix and with up to 30% GGBS, the strength increases, i.e., the pozzolanic reactions are encouraged, and cementation of the jarosite-GGBS mixture particles occurs. However, with further addition of the binder (GGBS), the OMC increases due to segregation of particles leading to a reduction in rate of pozzolanic reaction, which illustrates that the extra binder particles do not react and results in unbonded particles, thus, a decrease in the overall strength occurs. Similar observations have also been made by Bell [83], Kate [84], and Sharma and Sivapullaiah [85]. However, the overall strength of the stabilized jarosite-GGBS matrix with 40% GGBS content is still higher than that of untreated jarosite. Thus, based on the strength characteristics, the addition of up to 30% GGBS can be utilized to stabilize the jarosite, effectively and efficiently.

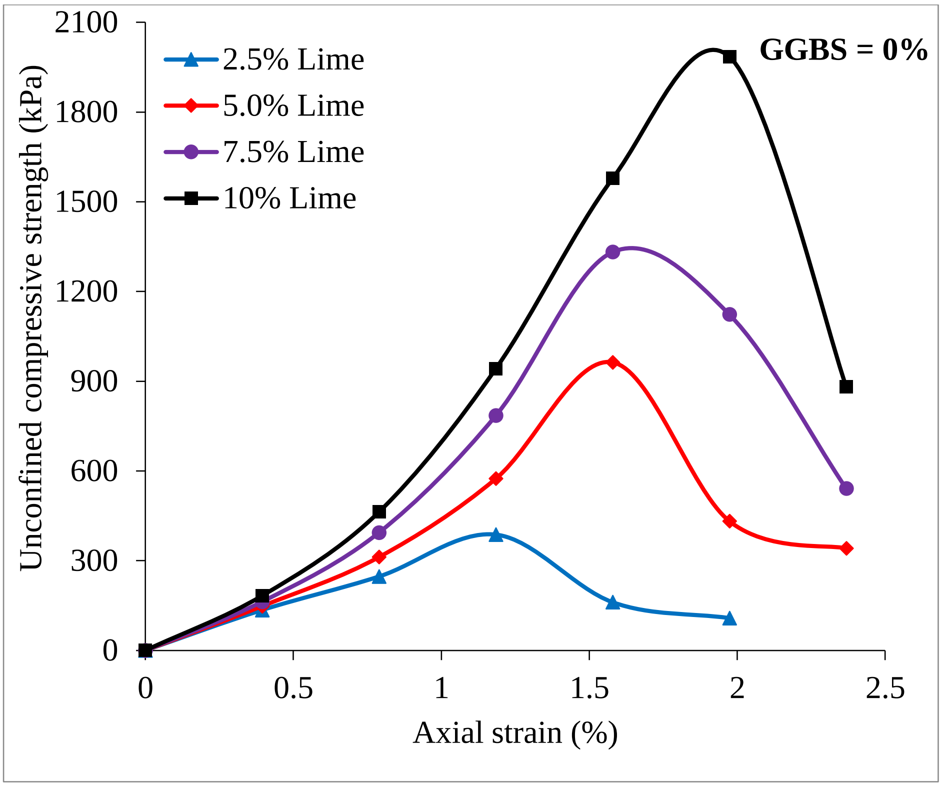
3.2.2 Effect of lime on jarosite-GGBS-lime mixture

The variations in the stress-strain curve of UCS and split tensile strength of jarosite with different percentages of GGBS and with the addition of lime (2.5 to 10% by weight of jarosite) and cured for 28 days are shown in Figures 3.7 and 3.8 respectively. In the absence of any pozzolanic chemical activator such as lime or cement, the GGBS indicates slightly self-hardening properties (Figure 3.7a & 3.8a). Similar observation has also been observed by Kaniraj and Havanagi [96].

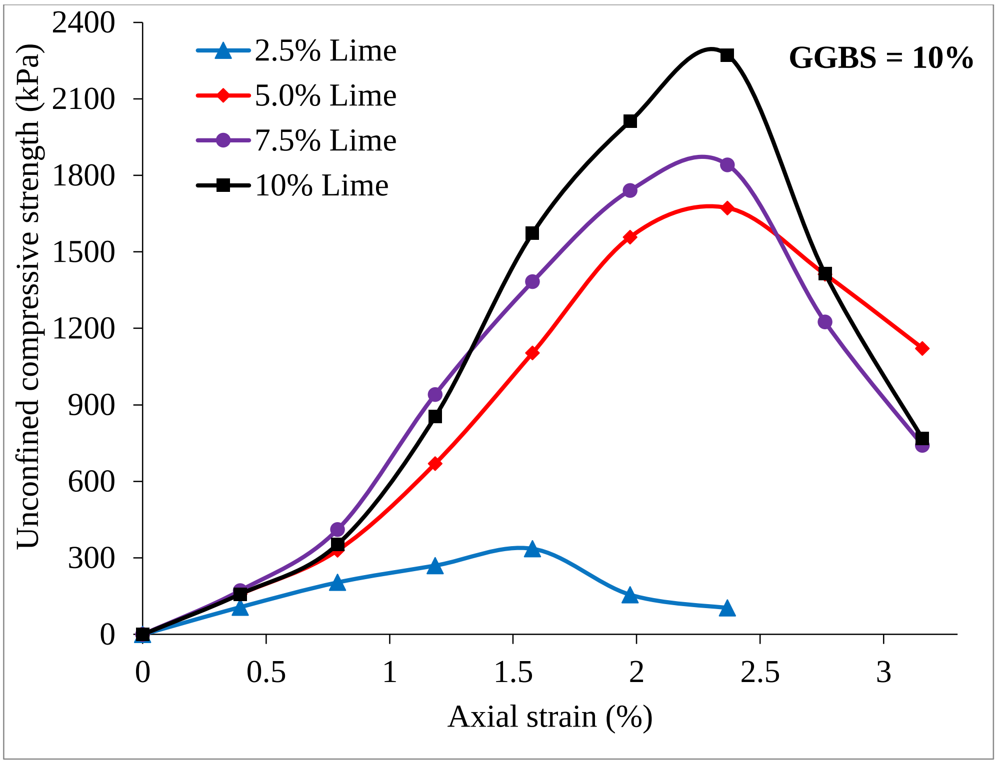
As apparent in Figure 3.6, it is clear that without any activator (lime), at 30% GGBS and 60 days curing period, the UCS increased from 167 to 622.87 kPa. Similarly, the split tensile strength increased from 42 to 143.28 kPa respectively. It is also clear from Figures 3.7 and 3.8 (b-e), that adding even a small amount of hydrated lime (2.5%), a significant improvement in the strength was observed. This result shows the critical role played through the adding of an activator (hydrated lime) with the pozzolanic material (GGBS). The combining of an activator (lime) boosts the chemical pozzolanic reaction. Thus the creation of a higher cemented composite provides extra strength to the jarosite-GGBS-lime blend.



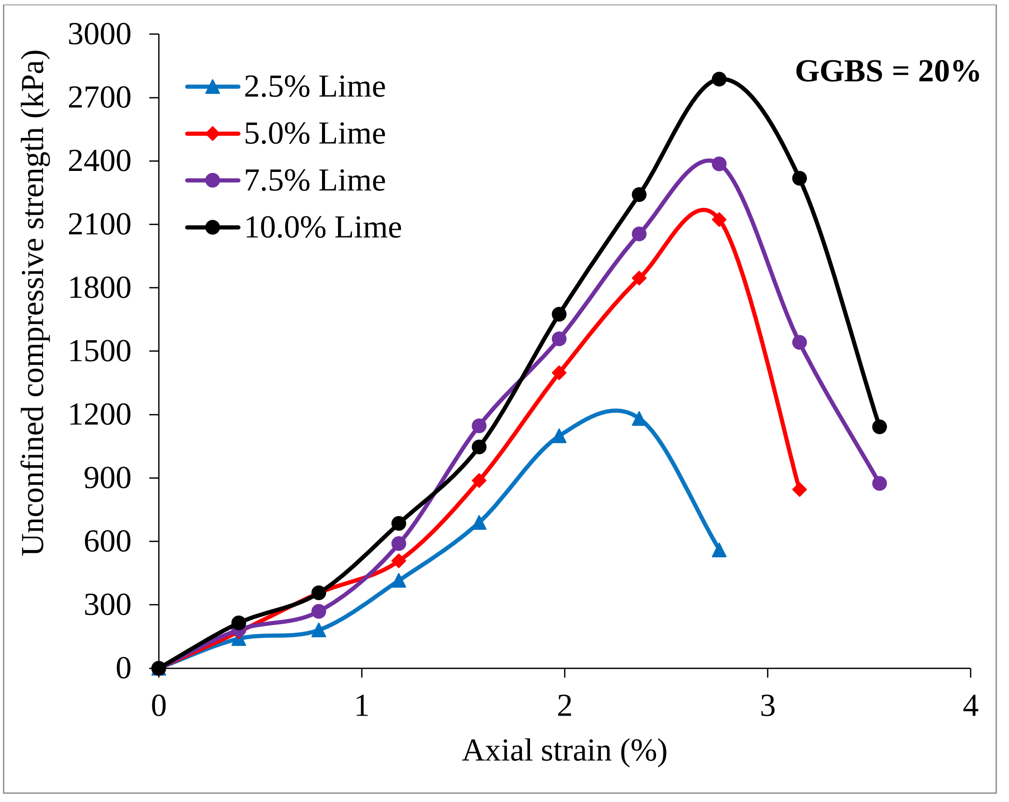
(a)


GGBS = 0%
2.5% Lime
5.0% Lime
7.5% Lime
10% Lime
Unconfined compressive strength (kPa)
Axial strain (%)
0
300
600
900
1200
1500
1800
2100
0
0.5
1
1.5
2
2.5

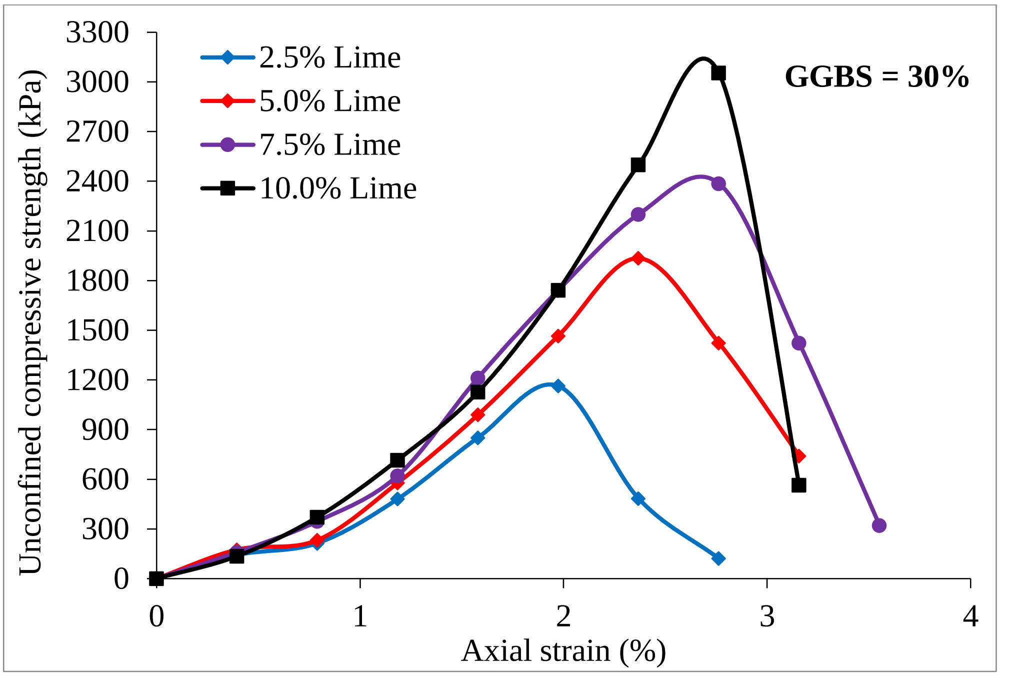
(b)


GGBS = 10%
2.5% Lime
5.0% Lime
7.5% Lime
10% Lime
Unconfined compressive strength (kPa)
Axial strain (%)
0
300
600
900
1200
1500
1800
2100
2400
0
0.5
1
1.5
2
2.5
3

(c)

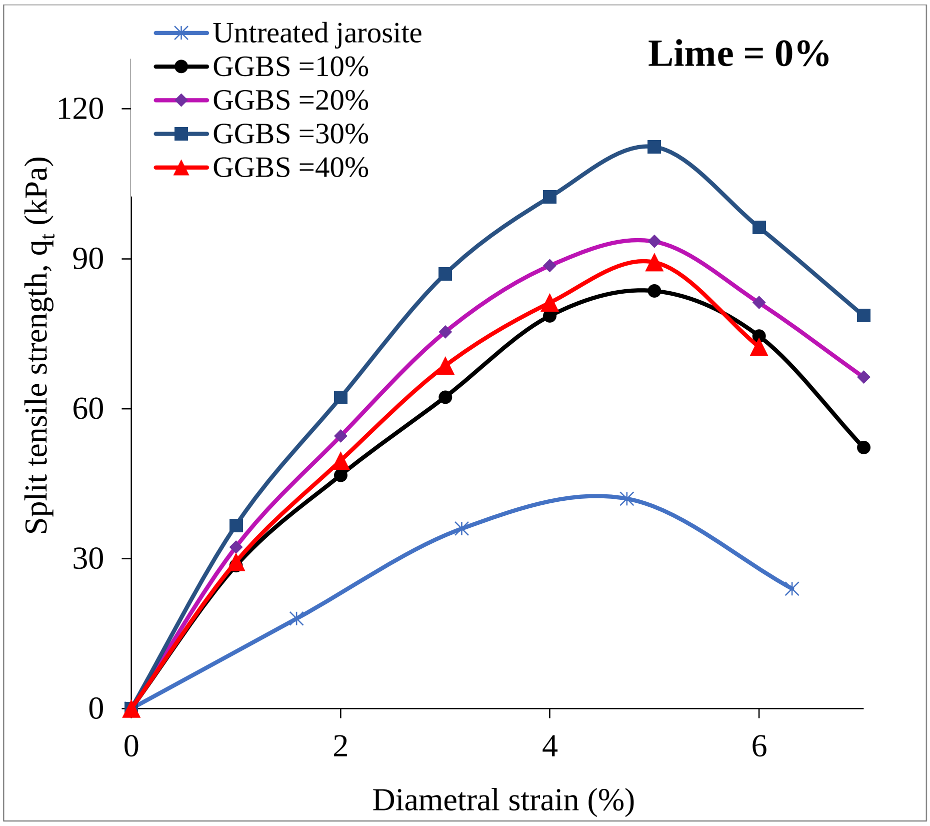


(d)



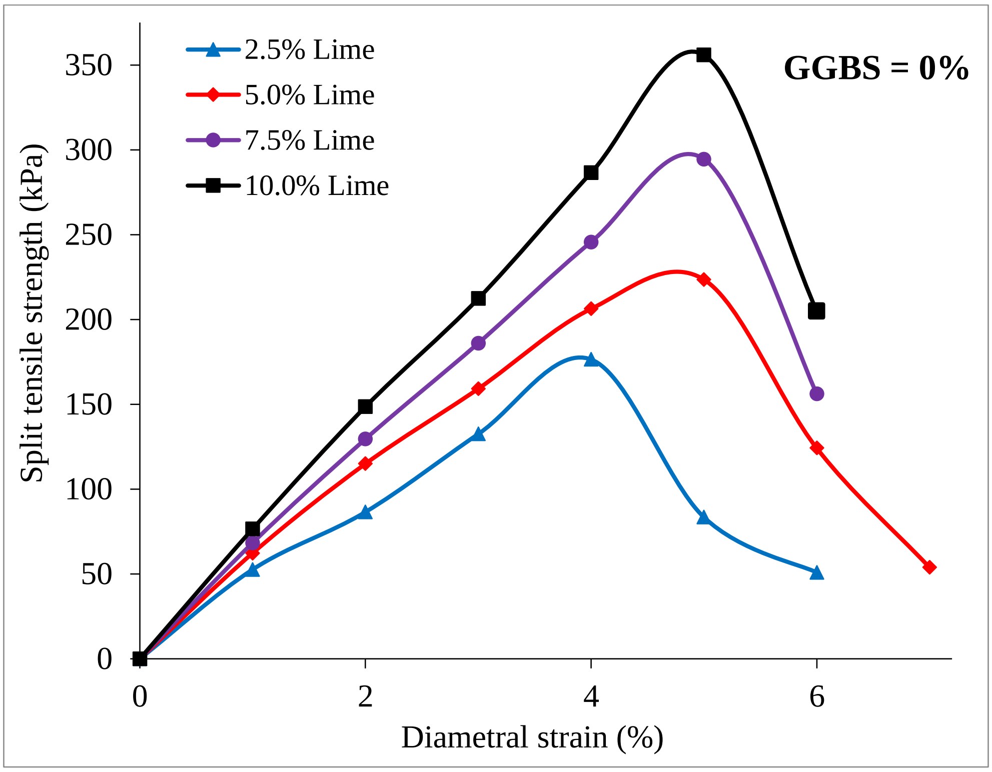
(e)

Figure 3.7 Effect of lime content on stress-strain behaviour of stabilized samples cured at 28 days during UCS test (a) 0% Lime (Jarosite-GGBS), (b) 0% GGBS, (b) 10% GGBS, (d) 20% GGBS & (e) 30% GGBS



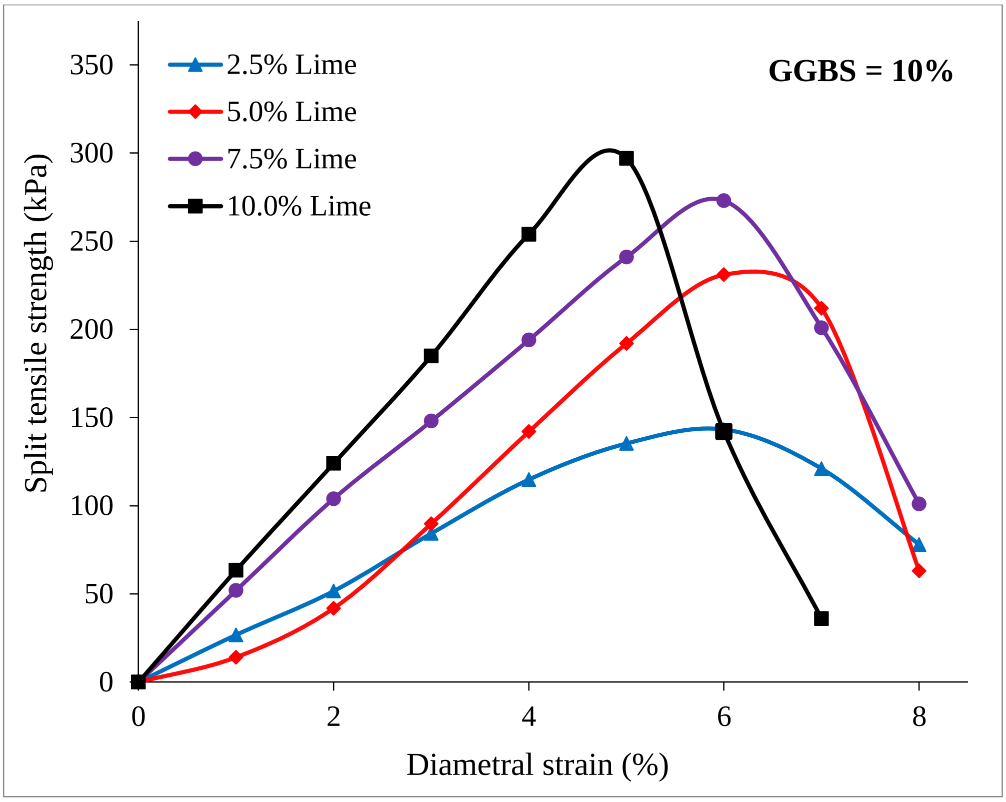
Untreated jarosite
GGBS =10%
GGBS =20%
GGBS =30%
GGBS =40%
Lime = 0%
Split tensile strength, q_t (kPa)
Diametral strain (%)
0
30
60
90
120
0
2
4
6

(a)

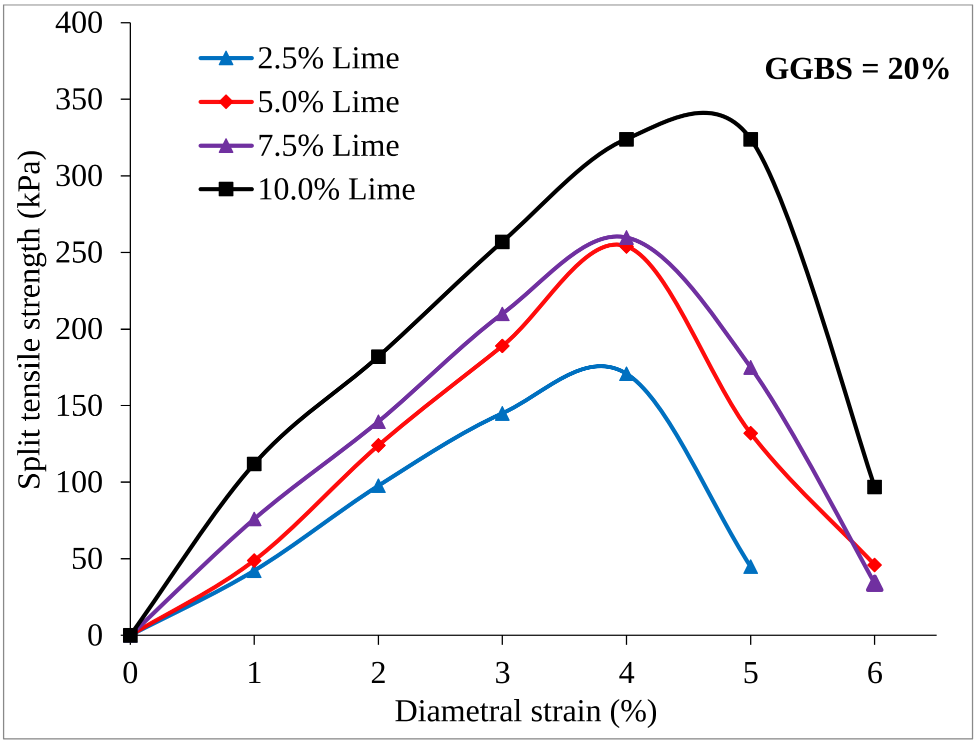


2.5% Lime
5.0% Lime
7.5% Lime
10.0% Lime
GGBS = 0%
Split tensile strength (kPa)
Diametral strain (%)
0
50
100
150
200
250
300
350
0
2
4
6

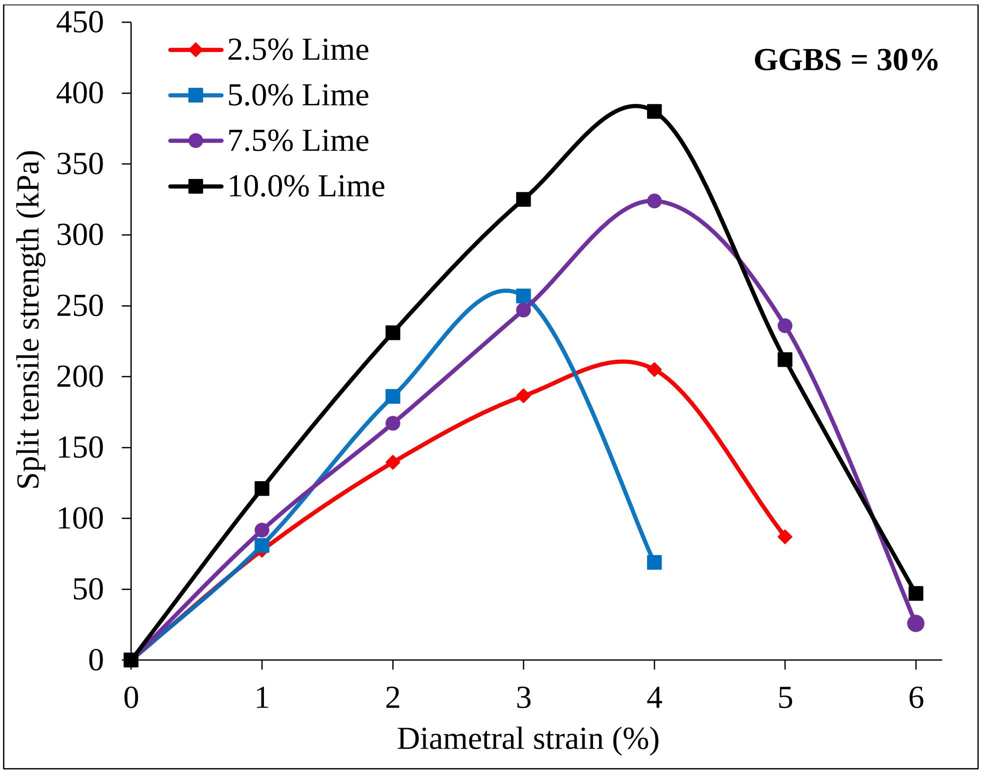
(b)


Split tensile strength (kPa)
350
300
250
200
150
100
50
0
2.5% Lime
5.0% Lime
7.5% Lime
10.0% Lime
GGBS = 10%
0
2
4
6
8
Diametral strain (%)

(c)


Split tensile strength (kPa)
400
350
300
250
200
150
100
50
0
2.5% Lime
5.0% Lime
7.5% Lime
10.0% Lime
GGBS = 20%
0
1
2
3
4
5
6
Diametral strain (%)

(d)



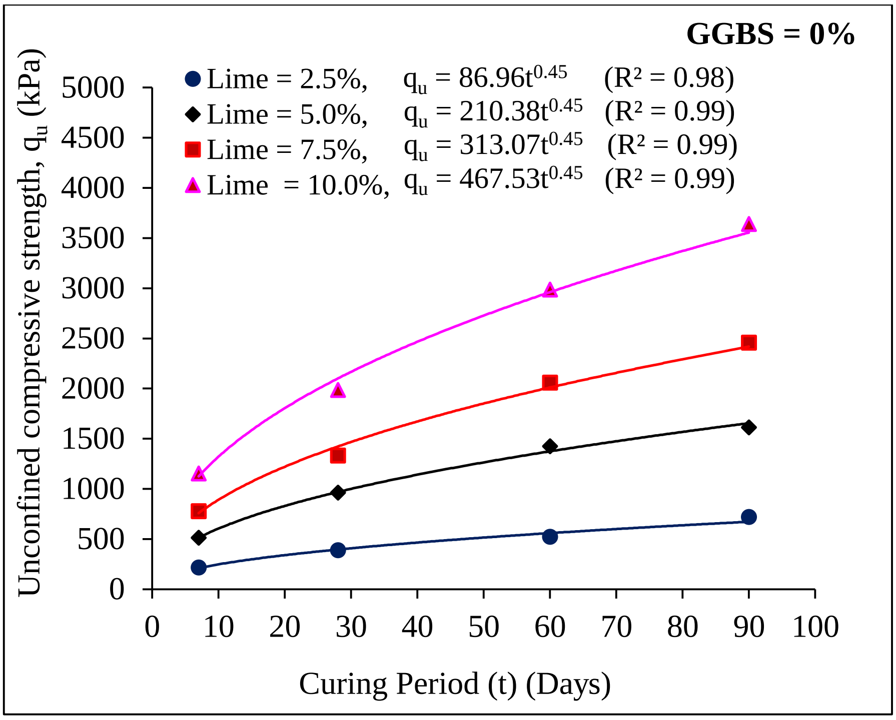
(e)

Figure 3.8 Effect of lime content on stress-strain behaviour of stabilized samples cured at 28 days during split tensile strength test (a) 0% Lime (Jarosite-GGBS), (b) 0% GGBS, (c) 10% GGBS, (d) 20% GGBS & (e) 30% GGBS

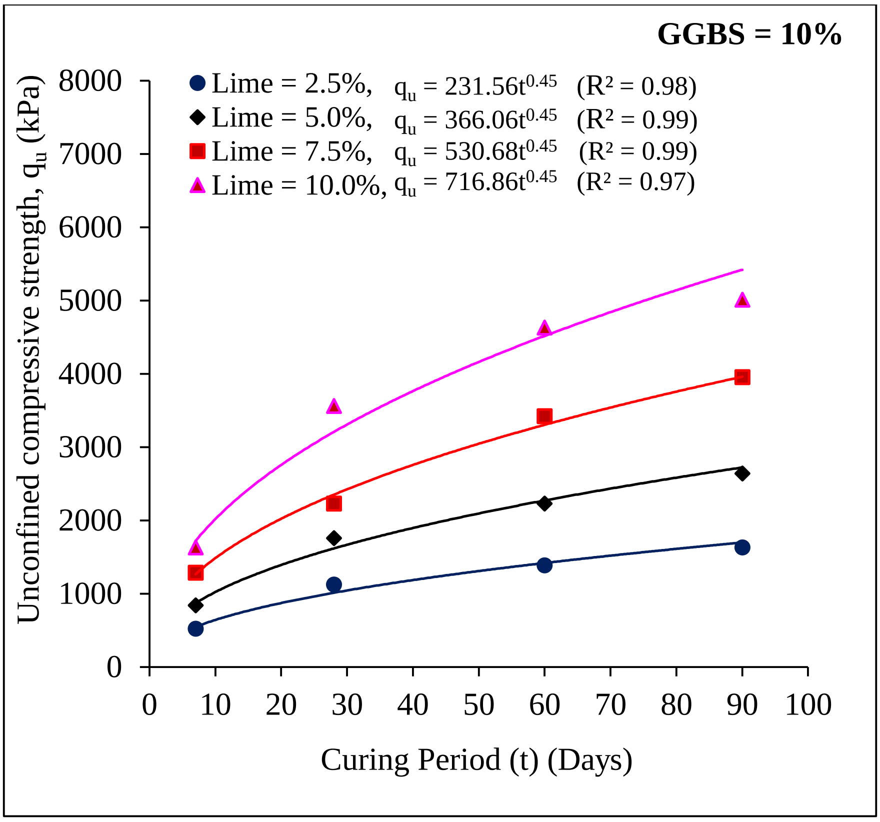
The past studies also validate that the addition of lime changes the particles composition, their mineralogy, physicochemical as well as the strength characteristics of stabilized fine-grained materials [86-87]. Also, it can be seen in Figures 3.7 and 3.8 that higher the curing period and lime content, higher is the brittleness of blended sample over untreated ones.

3.2.3 Relationship between unconfined compressive strength (q_u) and split tensile strength (q_t)

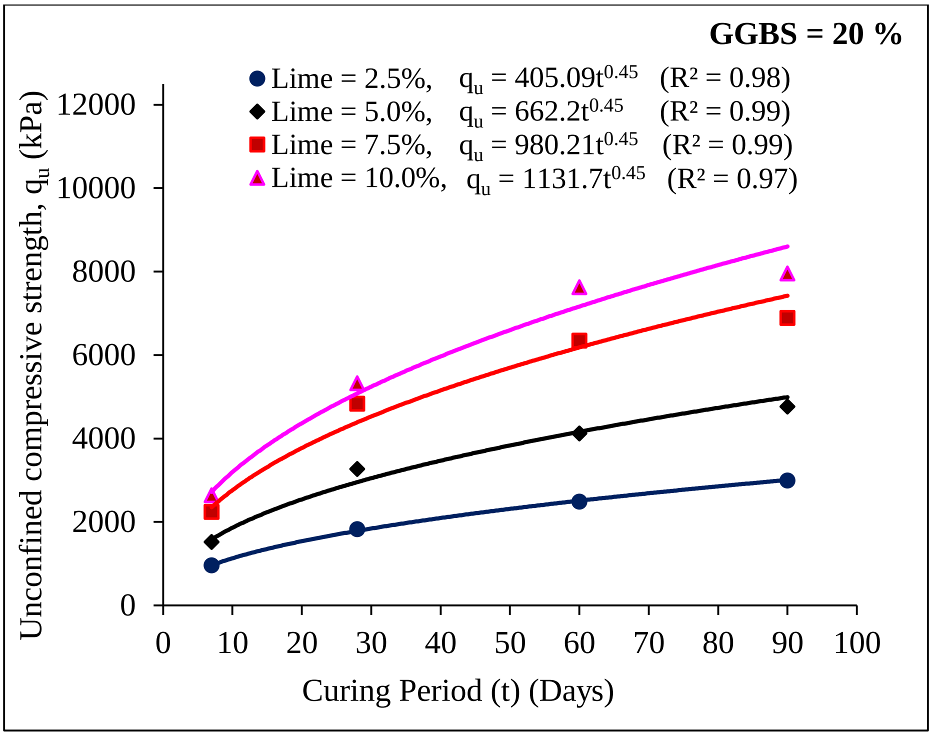
The UCS and split tensile strength of stabilized jarosite-GGBS-lime samples, formulated as a function of the adjusted curing period (t) and lime content (L), are shown in Figures 3.9 to 3.10 and 3.11 to 3.12 respectively.


GGBS = 0%
Lime = 2.5%, qu = 86.96t0.45 (R² = 0.98)
Lime = 5.0%, qu = 210.38t0.45 (R² = 0.99)
Lime = 7.5%, qu = 313.07t0.45 (R² = 0.99)
Lime = 10.0%, qu = 467.53t0.45 (R² = 0.99)
Unconfined compressive strength, qu (kPa)
Curing Period (t) (Days)

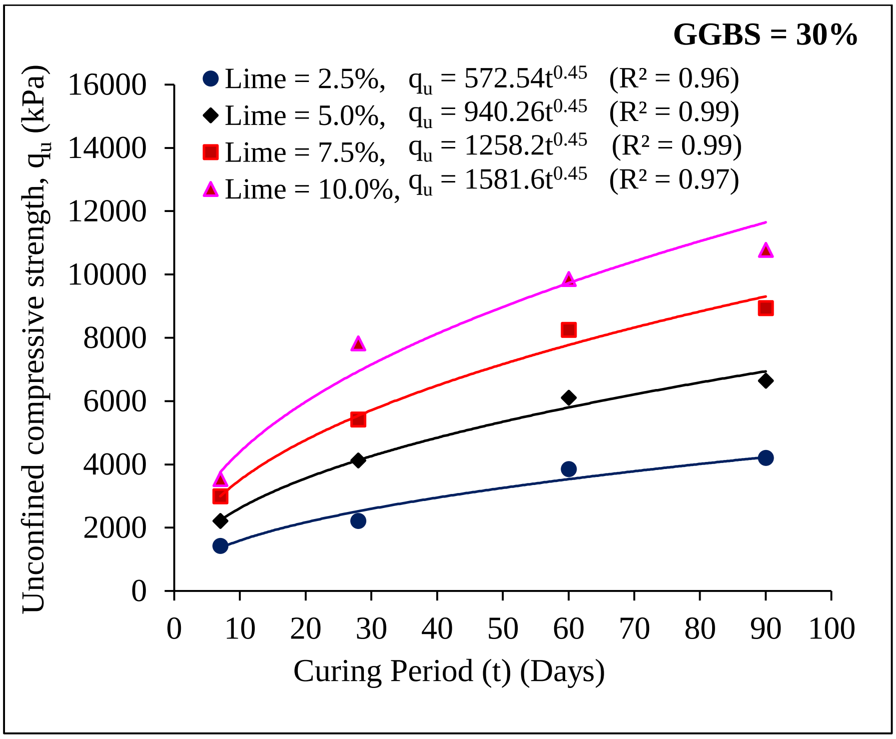
(a)


GGBS = 10%
Lime = 2.5%, qu = 231.56t0.45 (R² = 0.98)
Lime = 5.0%, qu = 366.06t0.45 (R² = 0.99)
Lime = 7.5%, qu = 530.68t0.45 (R² = 0.99)
Lime = 10.0%, qu = 716.86t0.45 (R² = 0.97)
Unconfined compressive strength, qu (kPa)
Curing Period (t) (Days)

(b)



(c)



(d)

Figure 3.9 Variation in UCS, q_u with various lime content, GGBS content and curing period: (a) 0% GGBS (jarosite-lime), (b) 10% GGBS, (c) 20% GGBS, and (d) 30% GGBS

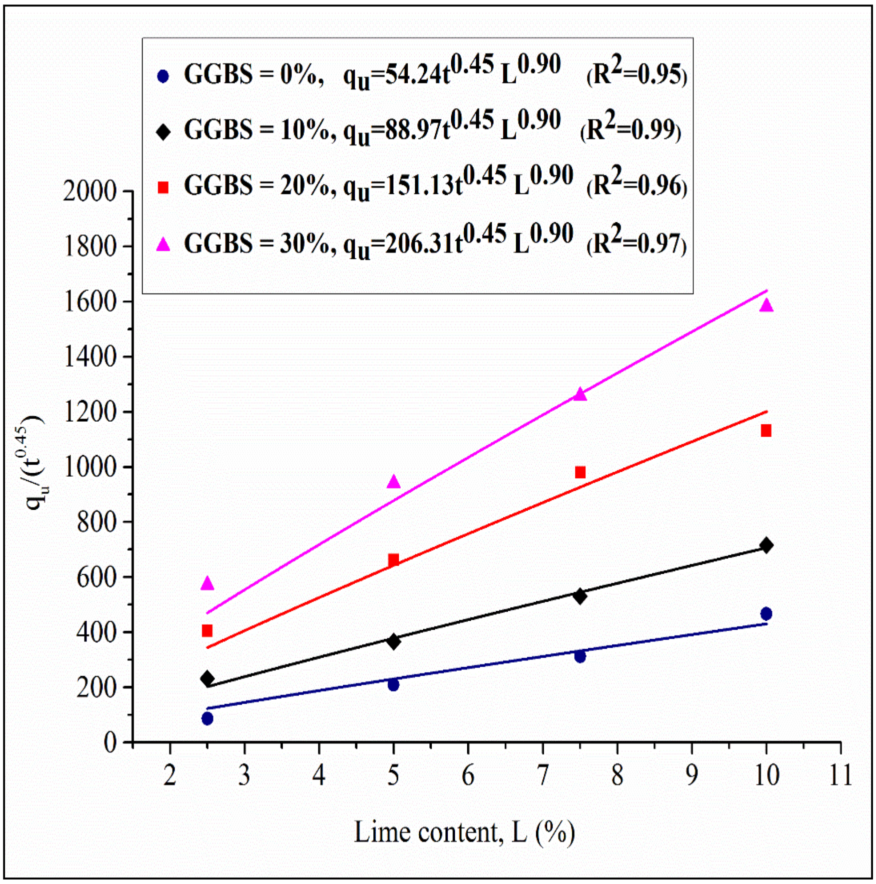
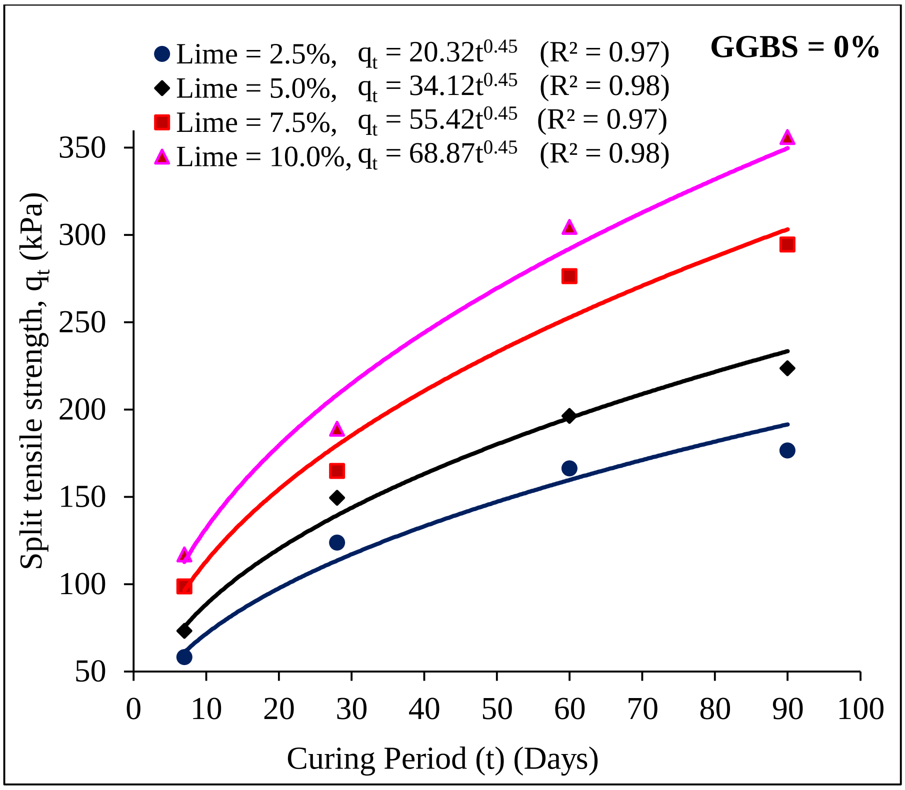


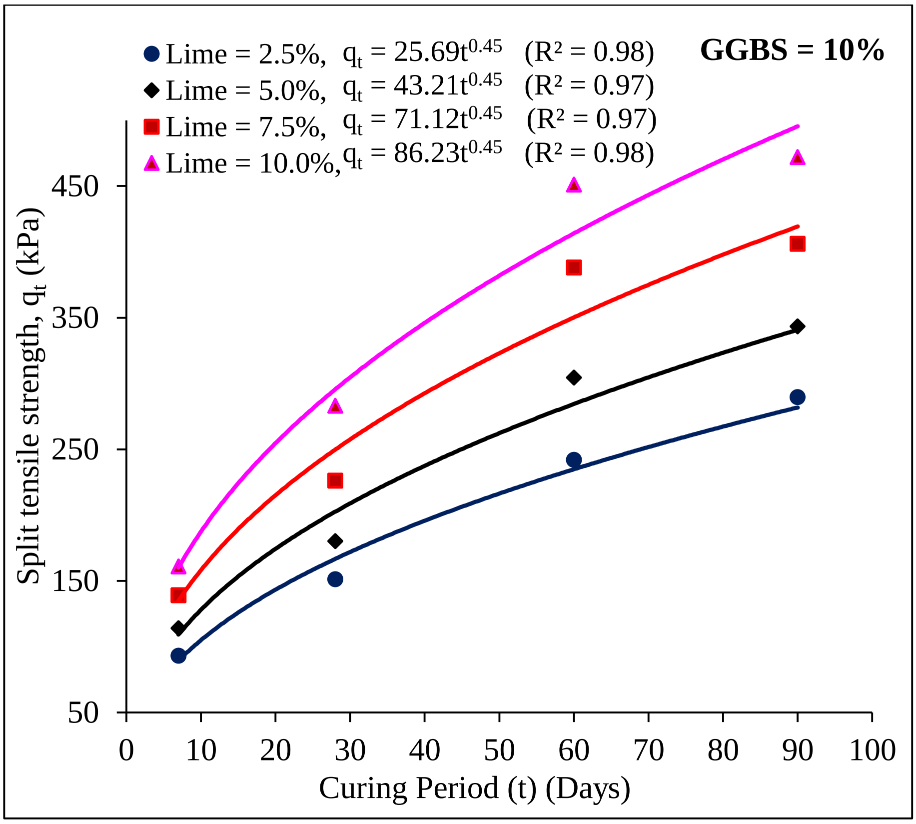
Figure 3.10 Relationship among q_u, t and L for various GGBS content

On observation of these Figures, it can be said that these two parameters, i.e., adjusted curing period (t) and lime content (L) are useful in normalizing results for jarosite-GGBS-lime mixtures. An attempt has been made to derive relationship among strengths (q_u and q_t), curing period (t), and lime content (L) for jarosite-GGBS-lime mixture with 0, 10, 20 and 30% GGBS (G).



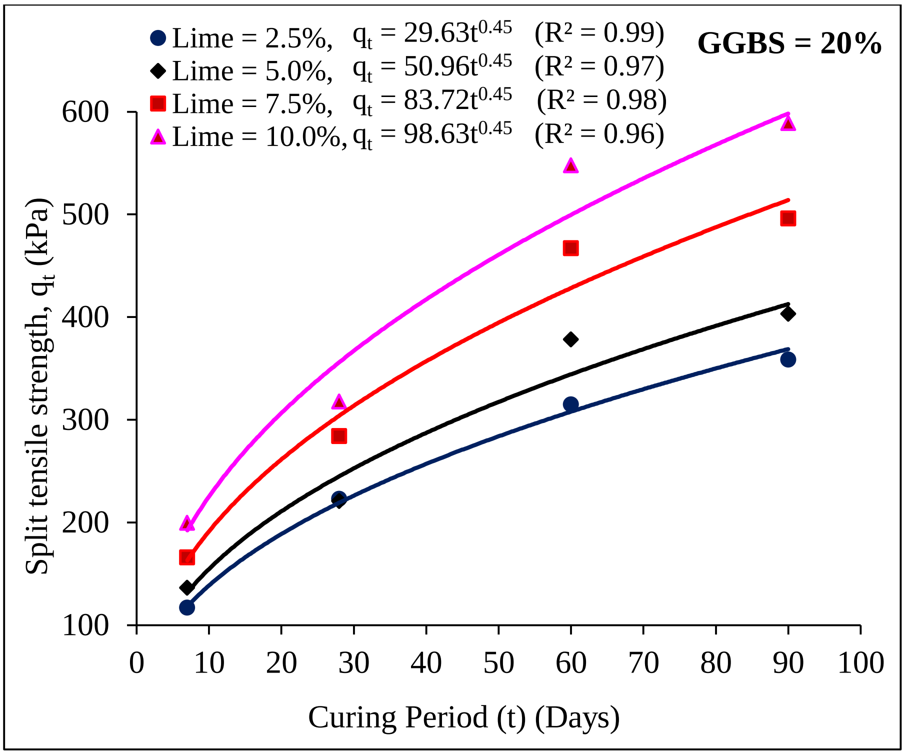
Lime = 2.5%, q_t = 20.32t^0.45 (R² = 0.97)
Lime = 5.0%, q_t = 34.12t^0.45 (R² = 0.98)
Lime = 7.5%, q_t = 55.42t^0.45 (R² = 0.97)
Lime = 10.0%, q_t = 68.87t^0.45 (R² = 0.98)
GGBS = 0%
Split tensile strength, q_t (kPa)
Curing Period (t) (Days)

(a)

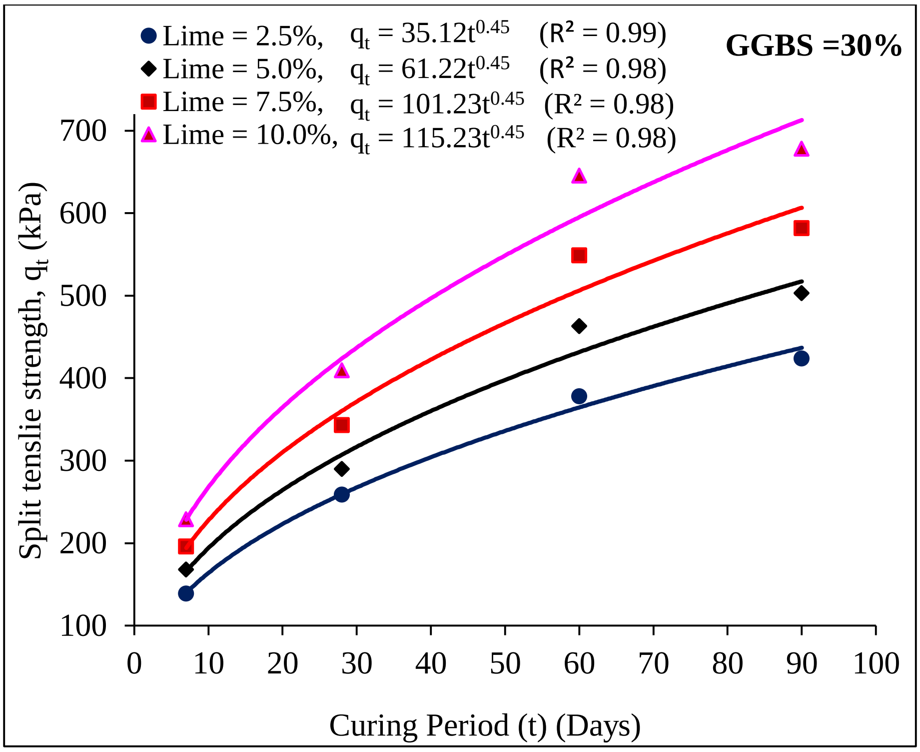


Lime = 2.5%, q_t = 25.69t^0.45 (R² = 0.98)
Lime = 5.0%, q_t = 43.21t^0.45 (R² = 0.97)
Lime = 7.5%, q_t = 71.12t^0.45 (R² = 0.97)
Lime = 10.0%, q_t = 86.23t^0.45 (R² = 0.98)
GGBS = 10%
Split tensile strength, q_t (kPa)
Curing Period (t) (Days)

(b)



(c)



(d)

Figure 3.11 Variation in split tensile strength, q_t with various lime content, GGBS content and curing period: (a) 0% GGBS (jarosite-lime), (b) 10% GGBS, (c) 20% GGBS, and (d) 30% GGBS

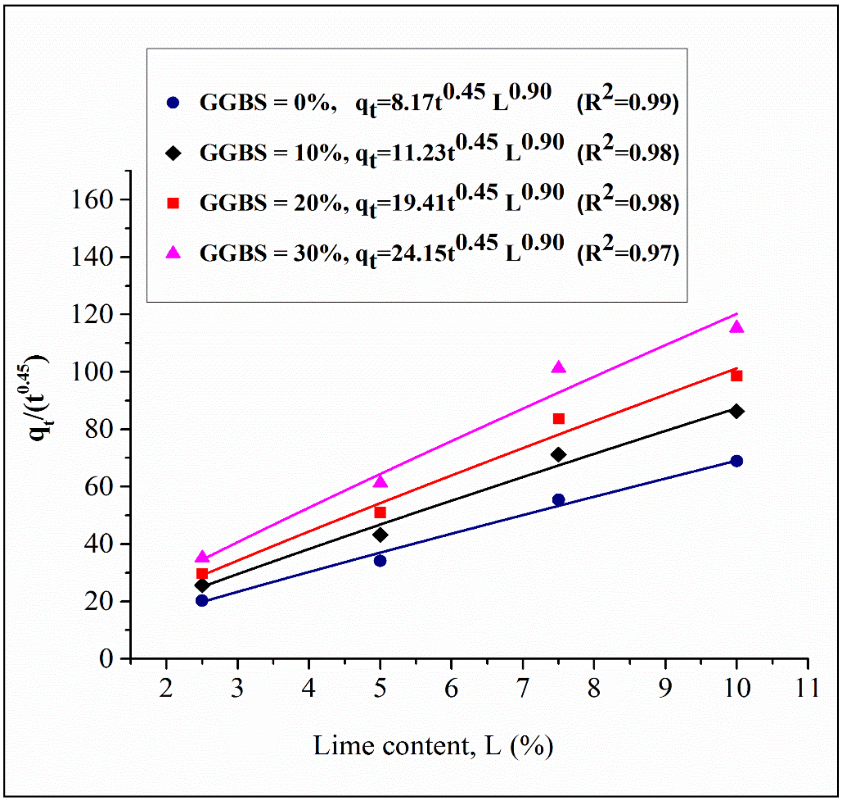


Figure 3.12 Relationship among q_t, t and L for various GGBS content

These relationships have been presented in Eqs. 3.1 to 3.4 for UCS (q_u) and 3.5 to 3.8 for split tensile strength (q_t) respectively. For easy comparison of results, the curing period (t) and lime content (L) have been normalized with power 0.45 and 0.90 respectively. In the relationships developed, good correlations have been observed varying between R^2 = 0.95 to 0.99.

Relations for unconfined compressive strength:

$$\text{For GGBS} = 0\%\,, q_u = 54.24\,(t)^{0.45}(L)^{0.90} \tag{3.1}$$

$$\text{For GGBS} = 10\%\,, q_u = 88.97\,(t)^{0.45}(L)^{0.90} \tag{3.2}$$

$$\text{For GGBS} = 20\%\,, q_u = 151.13\,(t)^{0.45}(L)^{0.90} \tag{3.3}$$

$$\text{For GGBS} = 30\%\,, q_u = 206.31\,(t)^{0.45}(L)^{0.90} \tag{3.4}$$

Relations for split tensile strength:

$$\text{For GGBS} = 0\%\,,\, q_t = 8.17\,(t)^{0.45}(L)^{0.90} \tag{3.5}$$

$$\text{For GGBS} = 10\%\,,\, q_t = 11.23\,(t)^{0.45}(L)^{0.90} \tag{3.6}$$

$$\text{For GGBS} = 20\%\,,\, q_t = 19.41\,(t)^{0.45}(L)^{0.90} \tag{3.7}$$

$$\text{For GGBS} = 30\%\,,\, q_t = 24.15\,(t)^{0.45}(L)^{0.90} \tag{3.8}$$

From above equations, it is advocated that variation in the strength of stabilized jarosite-GGBS-lime mixture depends directly on the curing period (t) and lime content (L). An adjusted power model fits well with a power of 0.45 for curing period (t) and 0.90 for lime content (L). It can also be observed that above equations have only a scalar variable, which is affected by the type of strength (unconfined compressive or split tensile) and GGBS content. Thus, unique relationships can be established among q_u or q_t with t, L, and G as shown in Figure 3.13 and Eqs. 3.9 and 3.10 respectively.

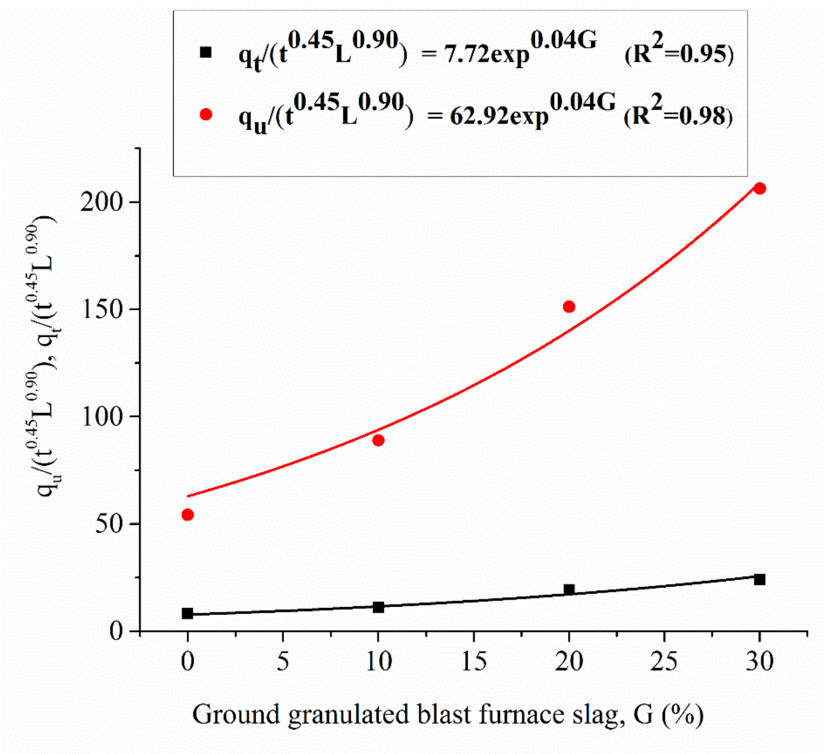


Figure 3.13 Relationship between unconfined compressive strength (q_u) and split tensile strength (q_t) with various lime content (L), GGBS content (G) and curing period (t)

$$q_u = 62.92\,(t)^{0.45}(L)^{0.90}exp^{0.04G} \tag{3.9}$$

$$q_t = 7.72\,(t)^{0.45}(L)^{0.90}exp^{0.04G} \tag{3.10}$$

Further, these two equations (3.9 and 3.10) have been shown as a single generalized equation [Eq. (3.11)], which can be utilized for determination of both unconfined compression strength (q_u) and split tensile strength (q_t).

$$q_{u \leftrightarrow} q_t\,(kPa) = A\,(t)^a(L)^b exp^{cG} \tag{3.11}$$

Where,

Factor A = a scalar coefficient which is influenced by the type of strength test, being 62.92 for UCS and 7.72 for split tensile strength, and

Factors a = 0.45, b = 0.90 and c = 0.04 are unique scalars for both tests.

Now, the ratio q_t/q_u can be written as (Eq. 3.12):

$$\frac{q_t}{q_u} = \check{C} = \frac{7.72\,(t)^{0.45}(L)^{0.90}exp^{0.04G}}{62.92\,(t)^{0.45}(L)^{0.90}exp^{0.04G}} = 0.122 \tag{3.12}$$

Hence, for jarosite, for all the range of lime content, GGBS content, and curing period studied, it may be concluded that the relationship, q_t/q_u (entitled as $\check{C}$) is unique [$\check{C}$ = 0.122] (Eq. 3.12). This parameter is independent of the curing period (t), lime content (L), and GGBS content (G). It is however advised that more studies are required for a higher degree of validation of these results with different types of materials such as soils, activators (binders), and for longer curing period.

3.2.3.1 Use of the equation developed

An illustration of calculation of a specific amount of lime (L), curing period (t), and GGBS (G) to reach a target value of q_u:

Target value of q_u = 3000 kPa and established curing period t = 28 days (most preferable). On the basis of availability and practical implication of GGBS, choose all four phases of GGBS content (G), i.e. 0, 10, 20 and 30%, and corresponding to these GGBS contents, find the appropriate amount of lime (L) to achieve a target value of q_u.

- Choose G = 0%, i.e. jarosite-lime blends

  Solution for L, obtained by Eq. (9) as L = 12.38%
- Choose G = 10%,

  Solution for L, obtained by Eq. (9) as L = 7.94%
- Choose G = 20%,

Solution for L, obtained by Eq. (9) as L = 5.09%

- Choose G = 30%,

Solution for L, obtained by Eq. (9) as L = 3.26%

By using the developed equation, the user/engineer can find appropriate lime content for desire strength (q_u), by fixing GGBS content and time period. Also, by considering q_u or $q_{t,}$ the other one can be easily found out by using Eq. 12.

3.3 Durability Study (Freezing and Thawing test)

The freezing and thawing test was conducted to assess the durability of stabilized jarosite-lime samples in accordance with ASTM D560M-15 [64]. This test method is used to determine the resistance of compacted jarosite-GGBS-lime matrix to repeated cycles of freezing and thawing (F-T) and also determine the optimum composition required to achieve a degree of adequate hardness to resist field weathering. For evaluation of durability of stabilized jarosite two types for studies namely as Strength and Weight loss (Material loss) were conducted and discussed subsequently.

3.3.1 Strength study

To observe the influence of lime and GGBS on durability, the stabilized jarosite samples were subjected to alternate cycles of freezing and thawing (0, 1, 3 and 5). The samples were then tested for unconfined compressive strength to find out the loss in strength. The effect of freezing-thawing cycles on the UCS of untreated jarosite waste and stabilized jarosite-GGBS-lime samples are shown in Figures. 3.14 and 3.15 respectively.

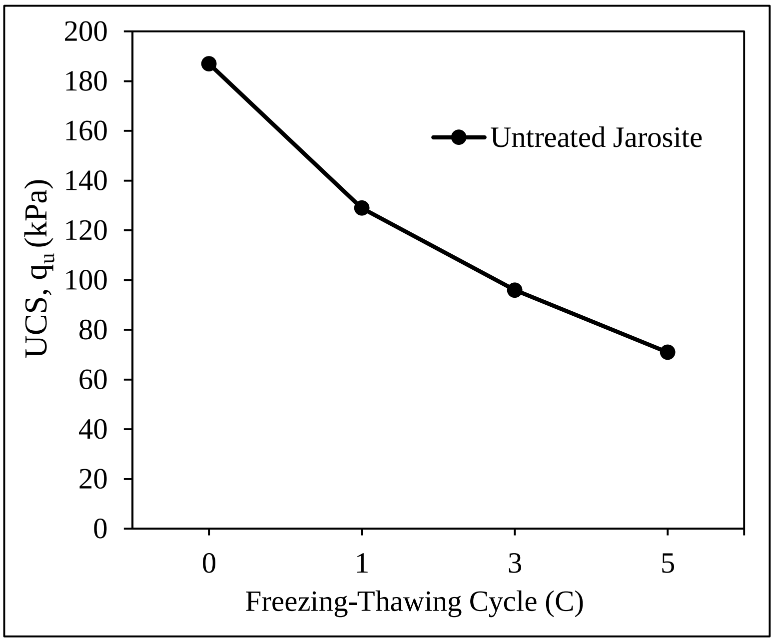
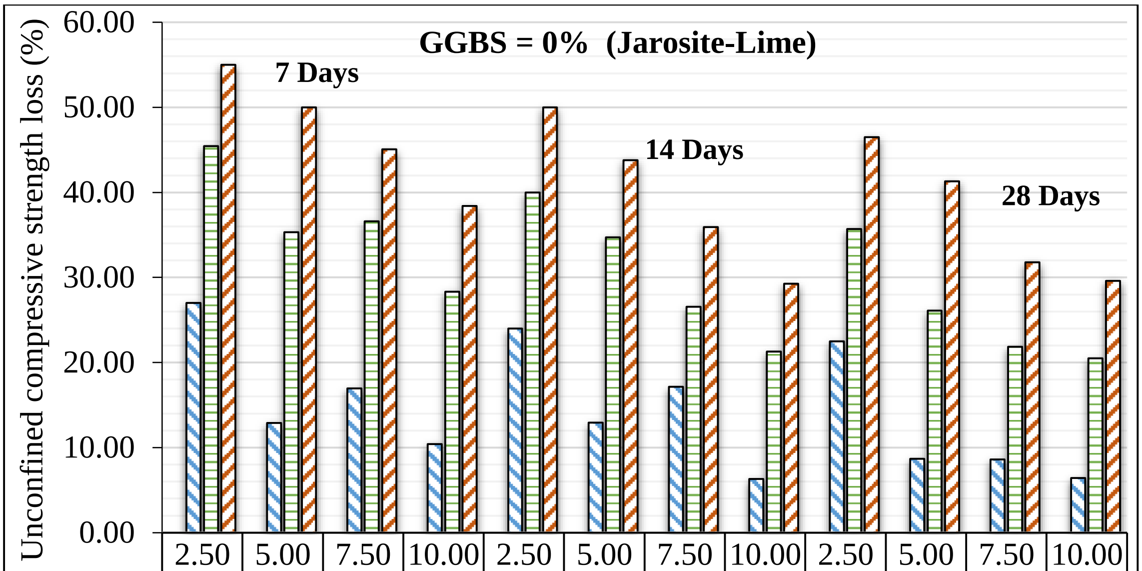
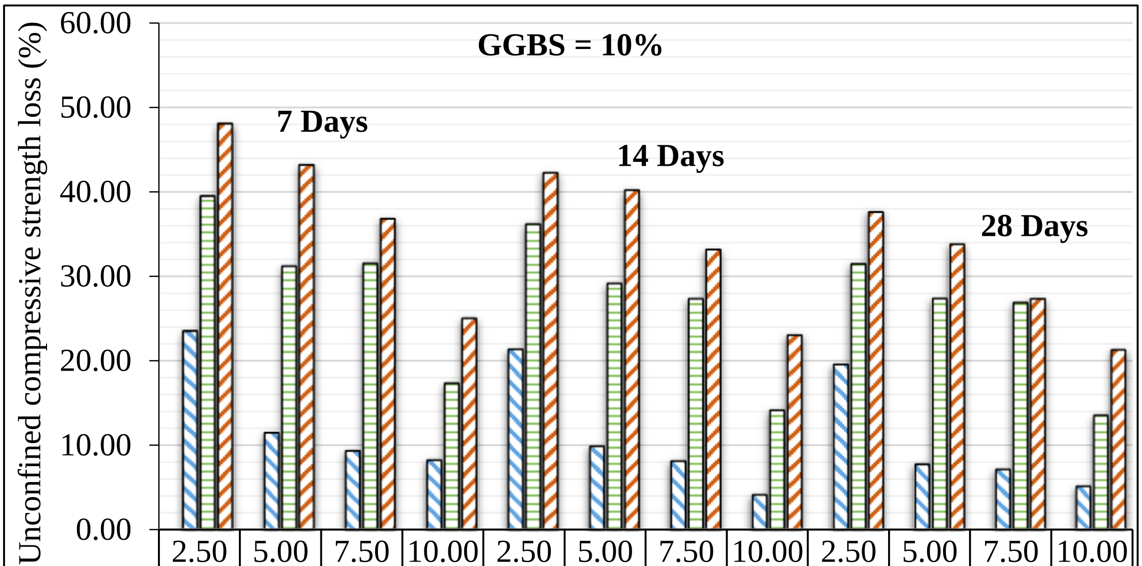


Figure 3.14 Variation in UCS (q_u) of untreated jarosite with freezing-thawing cycles



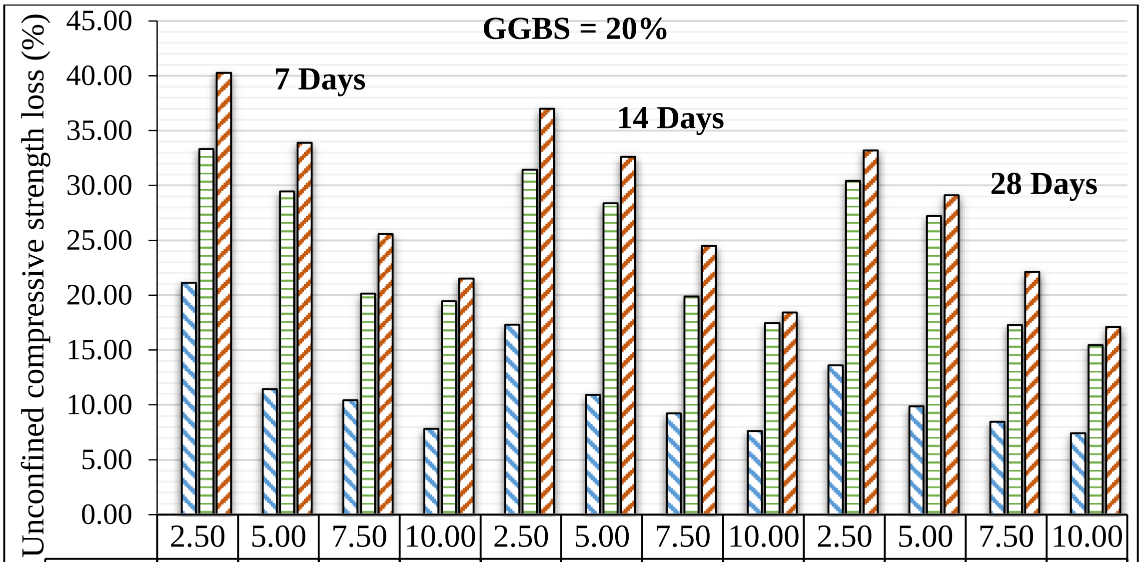
	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00
0 F-T	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1 F-T	27.00	12.90	16.96	10.41	24.00	12.94	17.14	6.33	22.50	8.70	8.61	6.42
3 F-T	45.45	35.32	36.61	28.33	40.00	34.70	26.56	21.28	35.71	26.09	21.85	20.50
5 F-T	55.00	50.00	45.08	38.41	50.00	43.77	35.94	29.25	46.50	41.30	31.79	29.61

(a)



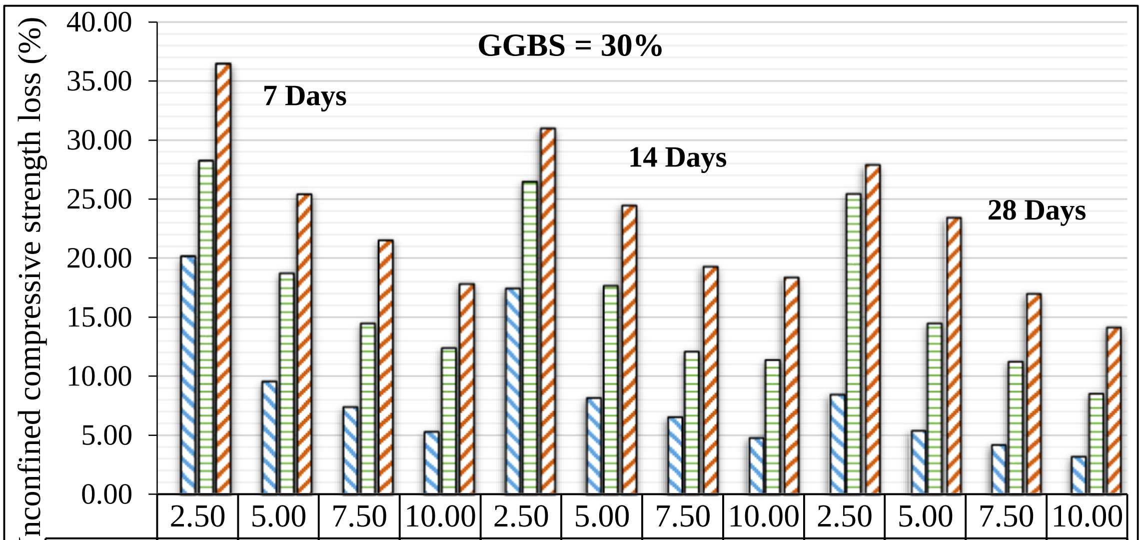
	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00
0 F-T	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1 F-T	23.51	11.54	9.39	8.32	21.36	9.92	8.17	4.18	19.59	7.81	7.19	5.19
3 F-T	39.52	31.21	31.54	17.35	36.18	29.18	27.36	14.19	31.50	27.41	26.89	13.56
5 F-T	48.07	43.21	36.87	25.12	42.27	40.23	33.21	23.15	37.67	33.87	27.42	21.41

(b)



	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00
0 F-T	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1 F-T	21.14	11.45	10.41	7.81	17.30	10.90	9.21	7.60	13.60	9.87	8.45	7.41
3 F-T	33.32	29.45	20.15	19.45	31.45	28.36	19.87	17.45	30.42	27.22	17.29	15.45
5 F-T	40.26	33.89	25.56	21.51	37.00	32.60	24.50	18.42	33.18	29.11	22.12	17.10

(c)



	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00	2.50	5.00	7.50	10.00
0 F-T	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1 F-T	20.11	9.56	7.40	5.32	17.42	8.15	6.55	4.80	8.45	5.39	4.21	3.22
3 F-T	28.23	18.74	14.50	12.43	26.45	17.68	12.12	11.41	25.44	14.52	11.29	8.57
5 F-T	36.45	25.44	21.55	17.88	31.00	24.48	19.33	18.42	27.89	23.45	17.02	14.20

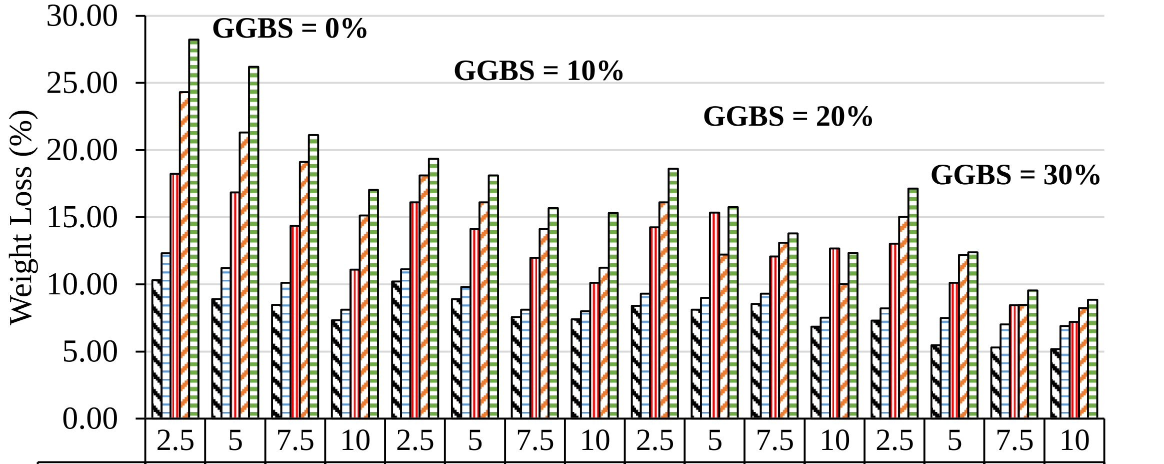
(d)

Figure 3.15 Variation in UCS of treated jarosite with F-T cycles and curing periods

Further, it was observed from the UCS results (Figure 3.15) of samples subjected to freezing and thawing that the addition of a higher percentage of GGBS and lime to jarosite plays a significant role in their durability. The addition of GGBS and lime considerably improves the freezing and thawing durability resistance as compared to untreated jarosite. The UCS values of untreated and stabilized jarosite samples are also affected by the increase in the number of cycles of freezing and thawing. For example, the reduction in unconfined compressive strength as a percentage loss, after five freezing-thawing cycles improved from 62% (untreated jarosite) to 29.61, 21.41, 17.10 and 14.20% at 0, 10, 20 and 30% GGBS content with 28 days curing period, 5 F-T cycles and 10% lime content. The variation in strength characteristics of untreated and stabilized samples, after freezing-thawing cycles, is due to the alteration occurring in the particle-to-particle arrangement that causes the weakening in the strength (Figure 3.19). Thus, it is likely that the deterioration happens in the pozzolanic bonds developed due to the reaction of the active/free aluminous and silicious minerals (in jarosite) and the calcium (in lime). Many researchers [88-90] have also reported similar findings.

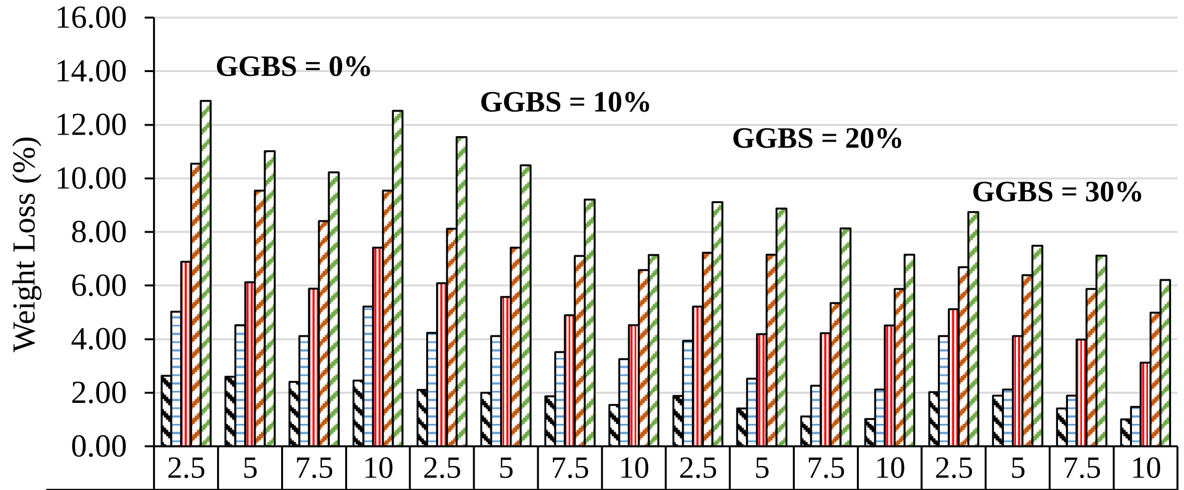
3.3.2 Weight loss (Material loss) study

To observe the influence of lime and GGBS on durability, identical to strength loss study, stabilized samples subjected to alternate Freeze-Thaw (0-5) were tested for material/ weight loss study. The variation in weight loss due to alternate F-T cycles is presented in Figure 3.16. It was revealed that an increase in GGBS content, lime content and curing period, the weight loss reduces. For example, at 0% GGBS, weight loss is 12.52%, which reduces to 7.15, 7.14 and 6.21% at 10, 20 and 30% GGBS content with 28 days curing period and 5 F-T cycles.



	2.5	5	7.5	10	2.5	5	7.5	10	2.5	5	7.5	10	2.5	5	7.5	10
1 F-T	10.	8.8	8.4	7.3	10.	8.9	7.5	7.4	8.4	8.1	8.5	6.8	7.3	5.4	5.2	5.1
2 F-T	12.	11.	10.	8.1	11.	9.8	8.1	7.9	9.3	9.0	9.3	7.5	8.2	7.4	7.0	6.8
3 F-T	18.	16.	14.	11.	16.	14.	11.	10.	14.	15.	12.	12.	13.	10.	8.4	7.2
4 F-T	24.	21.	19.	15.	18.	16.	14.	11.	16.	12.	13.	10.	15.	12.	8.4	8.2
5 F-T	28.	26.	21.	17.	19.	18.	15.	15.	18.	15.	13.	12.	17.	12.	9.5	8.8

(a)



	2.5	5	7.5	10	2.5	5	7.5	10	2.5	5	7.5	10	2.5	5	7.5	10
1 F-T	2.63	2.60	2.41	2.45	2.11	2.00	1.87	1.54	1.88	1.41	1.11	1.02	2.02	1.89	1.41	1.01
2 F-T	5.02	4.52	4.11	5.22	4.23	4.11	3.52	3.25	3.93	2.52	2.27	2.12	4.12	2.12	1.89	1.47
3 F-T	6.89	6.12	5.89	7.41	6.09	5.58	4.89	4.52	5.22	4.19	4.22	4.51	5.12	4.11	3.98	3.12
4 F-T	10.5	9.54	8.41	9.54	8.12	7.41	7.11	6.58	7.23	7.15	5.34	5.88	6.69	6.39	5.87	4.99
5 F-T	12.8	11.0	10.2	12.5	11.5	10.4	9.21	7.14	9.11	8.88	8.14	7.15	8.74	7.49	7.12	6.21

(b)

Figure 3.16 Variation in weight loss of treated jarosite with F-T cycles (a) 7 Days curing period; (b) 28 Days curing period

Table 3.2 illustrates the permissible limits of weight loss of stabilized material recommended by Portland Cement Association (1959) [90a]. The jarosite used for this study belongs to A-4 soil group, i.e. the permissible range of material loss should be less than 10%. On a closer look of Figure 3.16 (b), it can be advocated that the material loss of lime-jarosite blends (without GGBS) are not in the permissible limits. Whereas, after addition of GGBS (>10%), the treated materials exhibit material loss within the permissible limits. Thus, as per durability concern, lime treated jarosite materials (without GGBS) didn't pass the durability test. However, the GGBS-lime treated jarosite materials passed the durability test and are suitable to be used in civil engineering applications.

Table 3.2 Permissible limits for Weight/ Material Loss after the completion of Freeze-Thaw cycle [90a]

Soil Groups [90b]	Usual types of significant constituent material	Stabilized Material Loss (%)
A-1,A-3 A-2-4 and A-2-5	Gravels and Sand, Silty or Clayey Gravel	< 14%
A-2-6, A-2-7, A-4 and A-5	Silty sand and Silty Soils	< 10%
A-6 and A-7	Clayey Soils	< 7%

3.4 Mineralogical and Morphological Study

The samples from strength tests with different lime content, GGBS content and curing period were further examined for their mineralogical changes using X-ray diffraction

(XRD) test and their morphological changes by utilizing scanning electron microscope (SEM) and energy-dispersive X-ray spectroscopy (EDX).

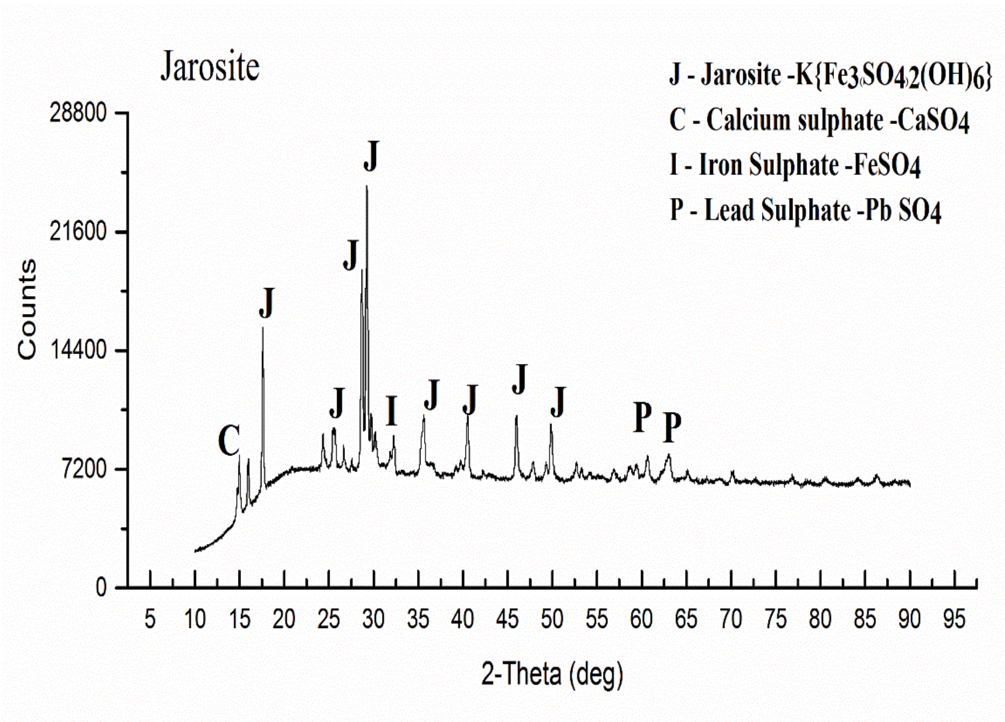
3.4.1 Mineralogical study (X-Ray diffraction)

As discussed in the methodology section, the X-Ray Diffraction (XRD) analysis was carried out by using high-resolution X-Ray Diffractometer- Rigaku Miniflex 600, Germany. The analysis of XRD graphs interprets that the untreated jarosite has major phases of Potassium Iron Sulphate Hydroxide (Jarosite) [$K\{Fe_3(SO_4)_2(OH)_6\}$]; Calcium sulfate ($CaSO_4$); Iron Sulphate ($FeSO_4$) and Lead Sulphate ($PbSO_4$). Similarly, GGBS has Quartz (SiO_2); Calcium oxide (CaO); Iron oxide (Fe_2O_3); and Aluminum oxide (Al_2O_3), (Figure 3.17 (a) & (b)). Whereas, after stabilization with GGBS and lime, the active/free aluminous and silicious minerals present in jarosite and GGBS reacts with calcium found in lime to produce calcium silicates hydrate [C-S-H] and calcium aluminates silicate hydrate [C-A-S-H] bonds (Figure 3.17 (c) &(d)). Others have reported similar findings [86, 91-92]. The following reactions can explain the formation of above cementitious compounds:

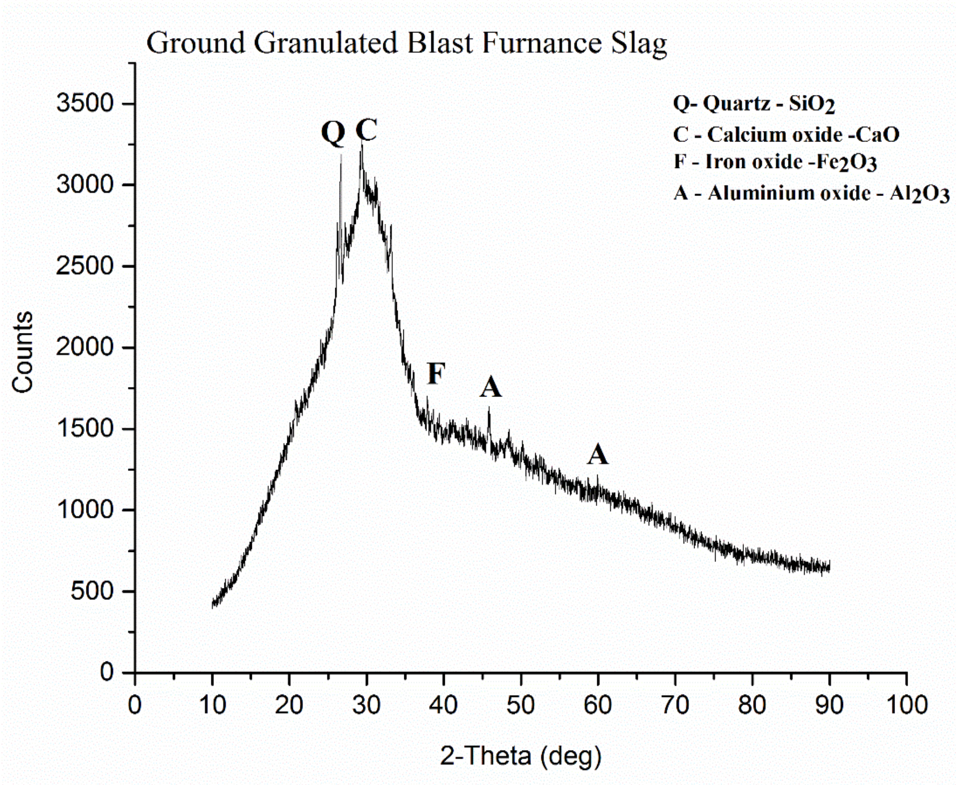
$$Ca(OH)_2 \text{ (ionization of hydrated lime)} \longrightarrow Ca^{2+} + 2(OH)^-$$

$$Ca^{2+} + (OH)^- + SiO_2 \text{ (soluble silica)} \longrightarrow \text{Calcium Silicate Hydrate}$$

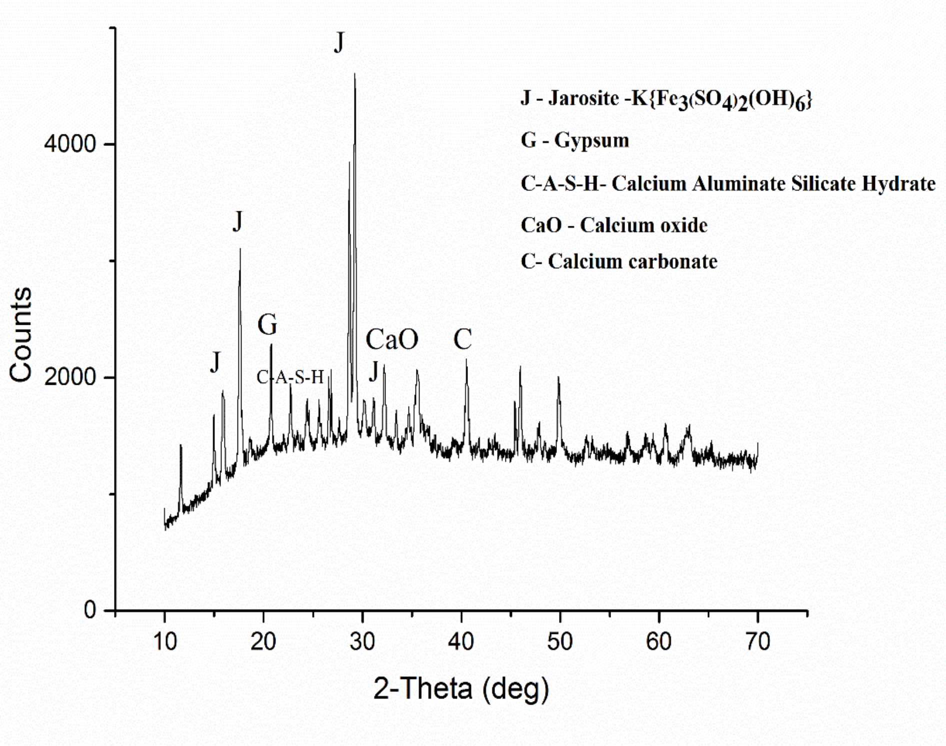
$$Ca^{2+} + (OH)^- + Al_2O_3 \text{ (soluble alumina)} \longrightarrow \text{Calcium Aluminates Hydrate}$$


Jarosite
J - Jarosite -K{Fe3(SO4)2(OH)6}
C - Calcium sulphate -CaSO4
I - Iron Sulphate -FeSO4
P - Lead Sulphate -Pb SO4
Counts
2-Theta (deg)

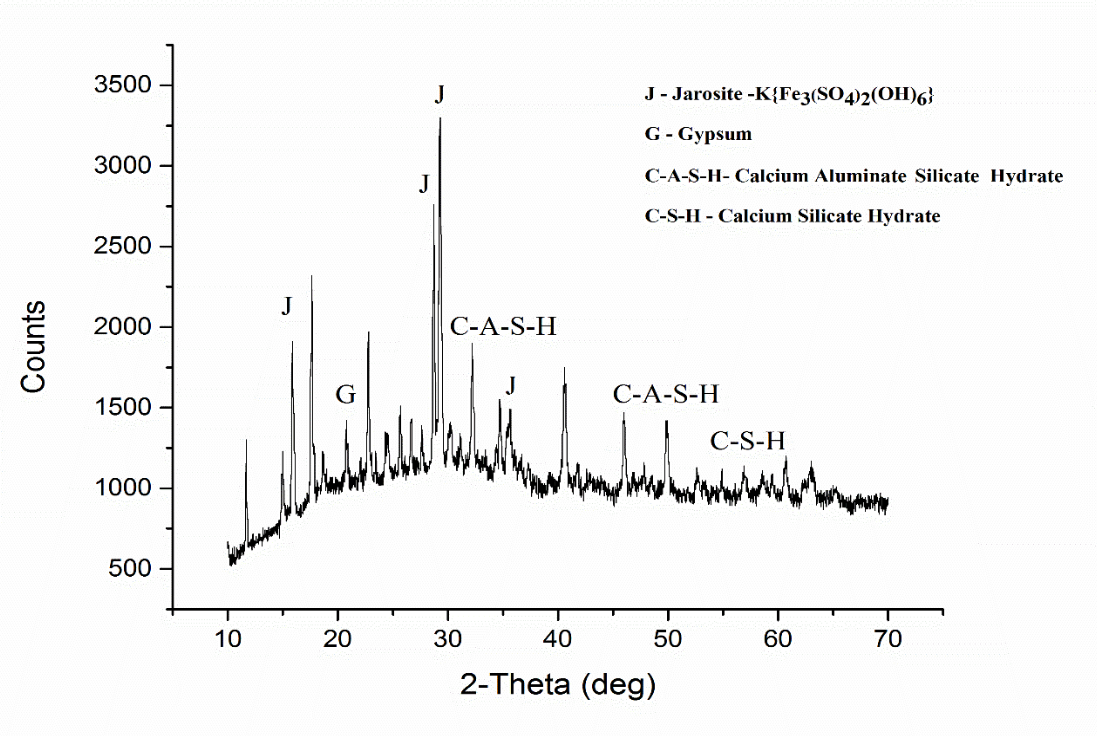
(a)


Ground Granulated Blast Furnance Slag
Q- Quartz - SiO2
C - Calcium oxide -CaO
F - Iron oxide -Fe2O3
A - Aluminium oxide - Al2O3
Counts
2-Theta (deg)

(b)



(c)



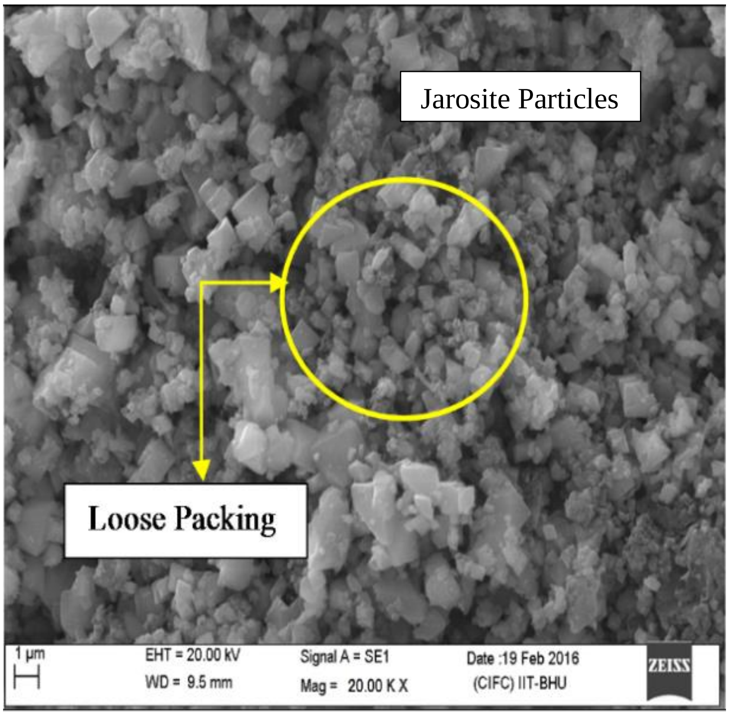
(d)

Figure 3.17 XRD images of (a) Jarosite; (b) GGBS; (c) Jarosite with 10% Lime (Jarosite with 0% GGBS); (d) Jarosite with 30% GGBS and 10% Lime

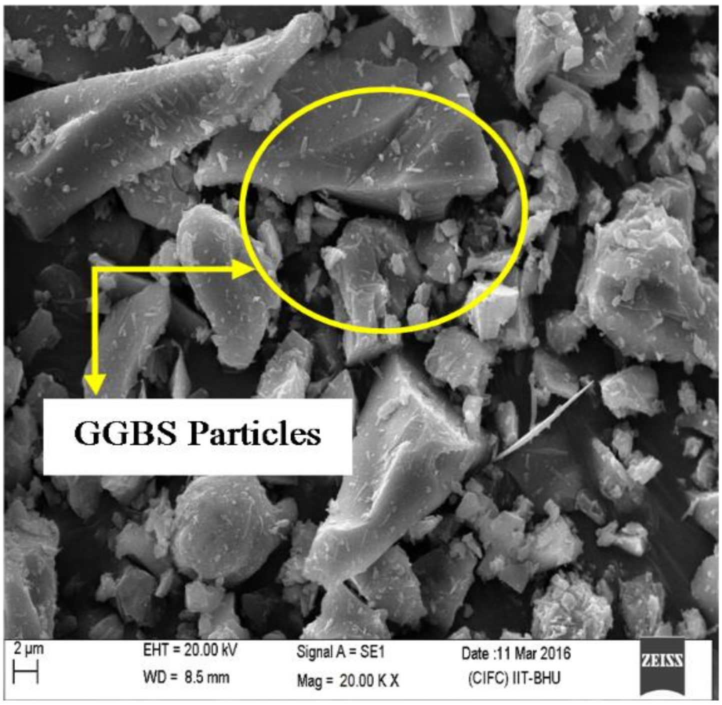
3.4.2 Morphological study (SEM-EDX)

The scanning electron microscope (SEM) corresponding to energy-dispersive X-ray spectroscopy (EDX) of various jarosite-GGBS-lime mixtures were carried out using high-resolution scanning electron microscope (SUPRA 40, Zeiss 4.0). For examining the change in the packing nature (due to pozzolanic reactions) of stabilized mixtures, several SEM images were recorded at different magnifications.

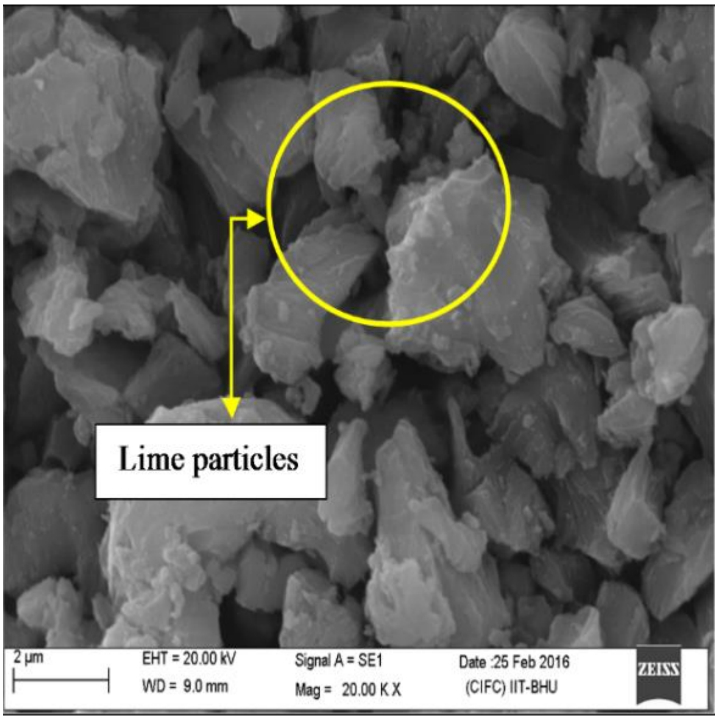
It was observed that the addition of lime to the jarosite-GGBS mixture produced a denser packing of particles as compared to untreated jarosite or jarosite-GGBS mixture. The denser packing of particles was obtained because of the active/free aluminous and siliceous minerals present in jarosite reacting with calcium found in lime to produce calcium silicates hydrate [C-S-H] and calcium aluminates hydrate [C-A-H] bonds, leading to agglomeration of particles. A typical SEM image of the jarosite-GGBS-lime mixture (Figure 3.18 (a), (b) & (c)) shows a loosely packed (porous) jarosite, GGBS and lime particles arrangement (uncemented) respectively. Further Figure 3.18 (d) shows jarosite-GGBS mixture with 30% GGBS content (0% lime) with 90 days curing period which indicating that closed and dense matrix (partial agglomeration) produced as compared to jarosite alone. Similarly Figure 3.18 (e) & (f) shows the effect of 10% lime on jarosite without GGBS before and after durability (freezing-thawing) respectively, and indicates that the presence of lime in jarosite produces closed and compacted matrix, which further remains relatively intact after durability. Figure 3.18 (g) & (h) illustrates the influence of lime on the GGBS-jarosite mixture with 30% GGBS content, 10% lime and 90 days curing period before and after durability respectively, which indicates that larger agglomeration of remains even after F-T effect of durability study.


Jarosite Particles
Loose Packing
1 µm
EHT = 20.00 kV
WD = 9.5 mm
Signal A = SE1
Mag = 20.00 K X
Date :19 Feb 2016
(CIFC) IIT-BHU
ZEISS

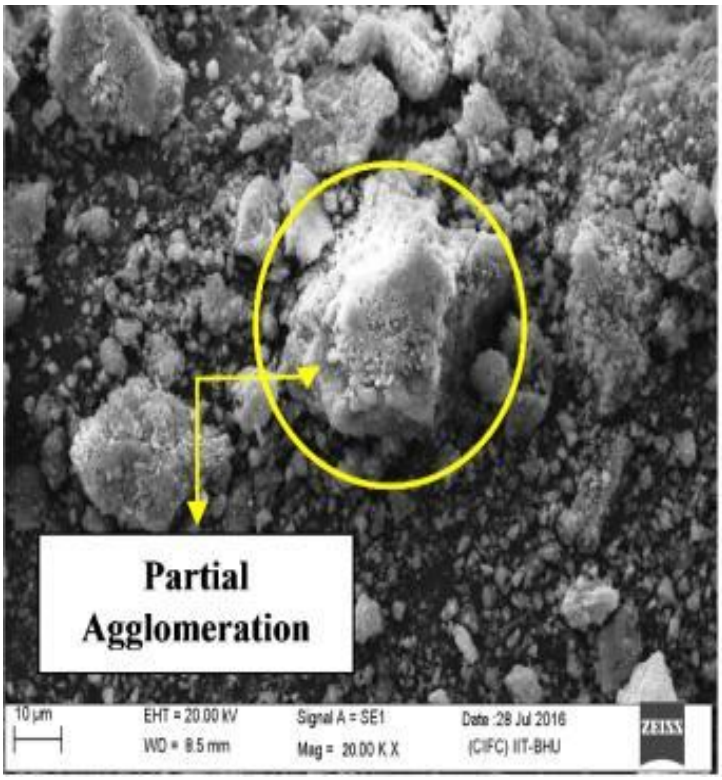
(a)


GGBS Particles
2 µm
EHT = 20.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 20.00 K X
Date :11 Mar 2016
(CIFC) IIT-BHU
ZEISS

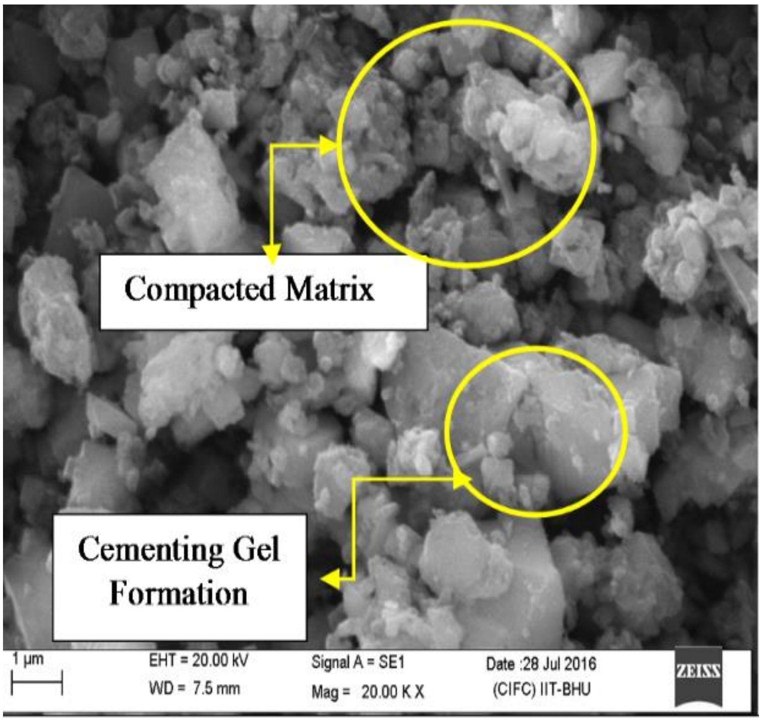
(b)


Lime particles
2 µm
EHT = 20.00 kV
WD = 9.0 mm
Signal A = SE1
Mag = 20.00 K X
Date :25 Feb 2016
(CIFC) IIT-BHU
ZEISS

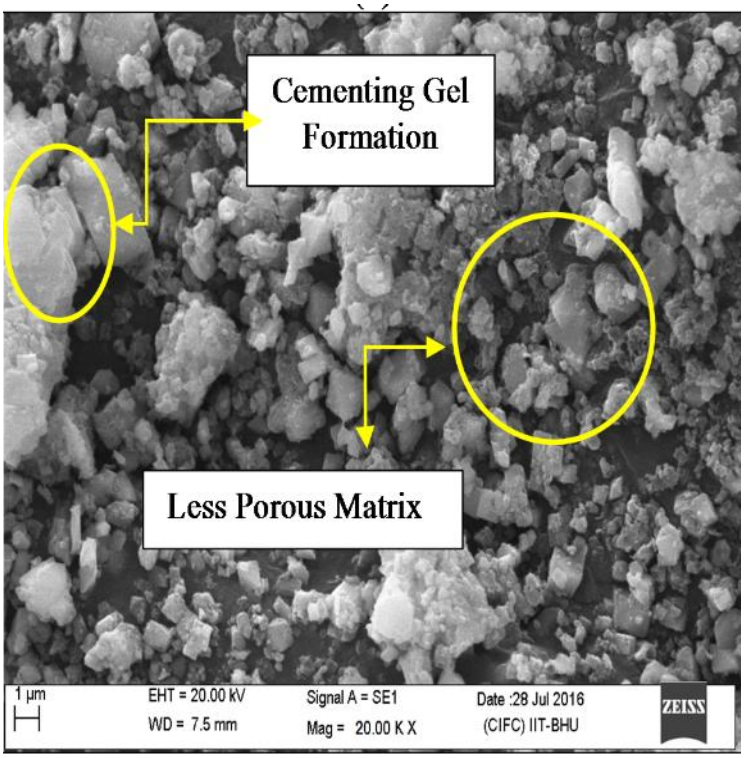
(c)


Partial
Agglomeration
10 µm
EHT = 20.00 kV
WD = 8.5 mm
Signal A = SE1
Mag = 20.00 K X
Date :28 Jul 2016
(CIFC) IIT-BHU
ZEISS

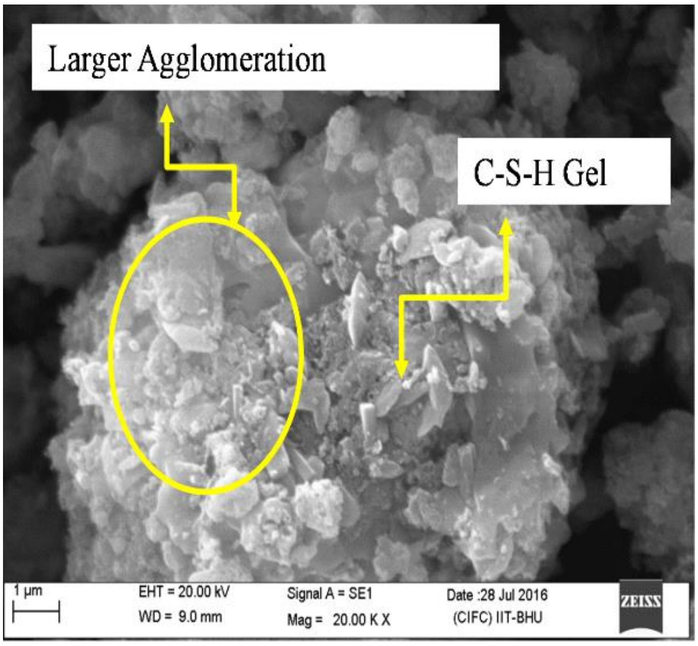
(d)


Compacted Matrix
Cementing Gel Formation
1 µm
EHT = 20.00 kV
WD = 7.5 mm
Signal A = SE1
Mag = 20.00 K X
Date :28 Jul 2016
(CIFC) IIT-BHU
ZEISS

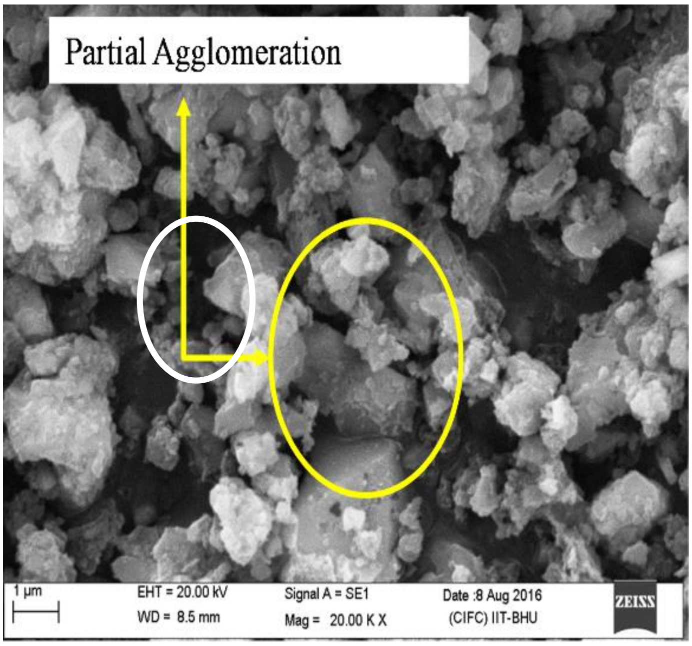
(e)


Cementing Gel Formation
Less Porous Matrix
1 µm
EHT = 20.00 kV
WD = 7.5 mm
Signal A = SE1
Mag = 20.00 K X
Date :28 Jul 2016
(CIFC) IIT-BHU
ZEISS

(f)



(g)



(h)

Figure 3.18 SEM images (Magnification = 20 kx) of jarosite-GGBS-lime mixture (a) jarosite; (b) GGBS; (c) Lime; (d) jarosite-GGBS mixture (30% GGBS at 90 days curing); (e) jarosite-lime mixture (10% lime at 90 days curing); (f) jarosite-lime mixture (after durability); (g) jarosite-GGBS-lime mixture (30% GGBS-10% lime at 90 days curing); (h) jarosite-lime-GGBS mixture (after durability)

Typical SEM-EDX images of selected mixtures of stabilized jarosite are shown in Figure 3.19. From SEM-EDX studies, it is concluded that the adding of lime in the GGBS-jarosite mixture enhances the strength characteristics due to larger agglomeration of particles. The changes in strength characteristics can be studied by the variation in Ca: Si and Si: Al ratios. Table 3.3 shows the variation in Ca: Si and Si: Al ratios of selected jarosite-GGBS-lime mixtures. From Table 3.3, it is observed that the adding of lime in jarosite-GGBS mixture with increasing GGBS content leads to a rise in Ca: Si ratio. The increase in Ca: Si ratio affirms the formation of a various cementing compound such as [C-S-H], [C-A-H] and [C-S-A-H], which are primarily involved in the enhancement of strength characteristics. Similar findings have also been reported by Kumar and Datta [97-98]. Furthermore, it was also seen that the adding of lime to GGBS-jarosite mixture with increasing GGBS content, the Si: Al ratio decreases, which also indicates the improvement in strength characteristics.

Table 3.3 Variation in Ca:Si and Si:Al ratio in various jarosite-GGSB-lime mixtures

Mixtures	Proportions (%)	Ca/Si	Si/Al
Jarosite	Untreated	0.56	0.79
Jarosite-GGBS	J+10% G	0.48	2.57
	J+30% G	1.09	0.87
Jarosite-GGBS-Lime	J+10% G+10% L	2.02	1.84
	J+20% G+10% L	3.75	1.80
	J+30% G+10% L	4.46	1.70

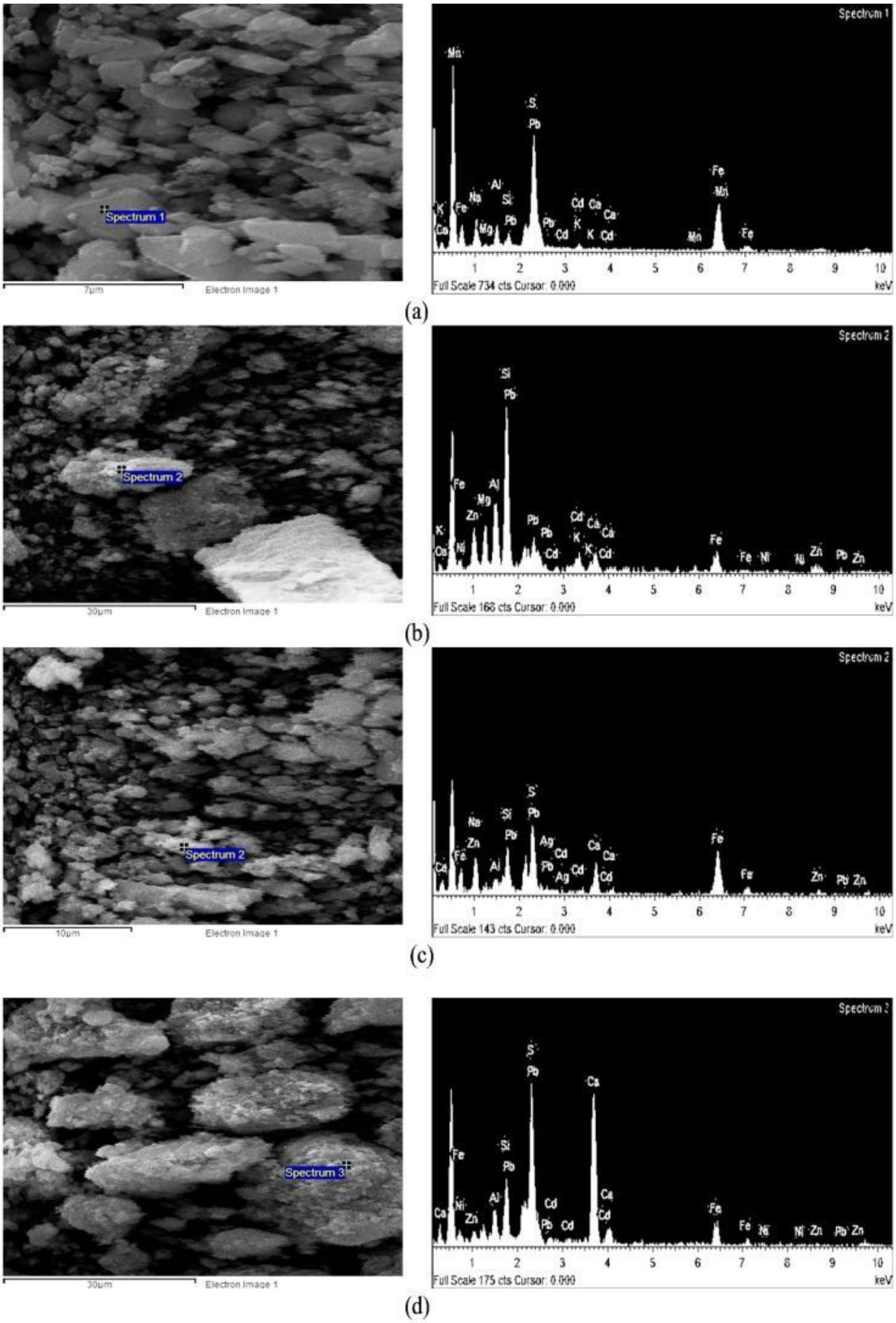


Figure 3.19 SEM-EDX images of (a) jarosite; (b) jarosite-GGBS mixture (30 % GGBS at 90 days curing); (c) jarosite-lime mixture (10% lime at 90 days curing); (d) jarosite-GGBS-lime mixture (30 % GGBS at 10% lime at 90 days curing.

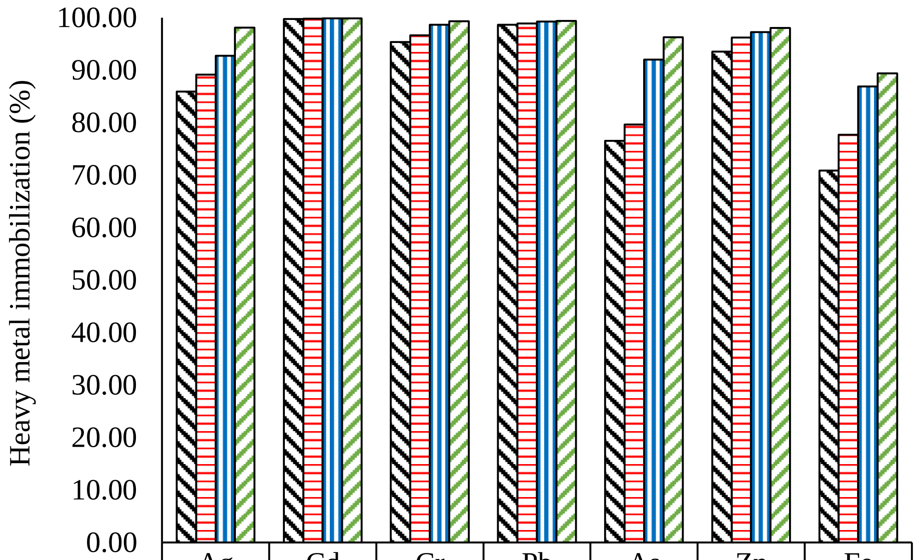
3.5 Toxicity Leachate Characteristics Procedure (TCLP) Study

For evaluation of the heavy metals and toxic elements, the United States Environmental Protection Agency (USEPA)-Toxicity Leachate Characteristics Procedure (TCLP) [25] was adopted. In the current study, Thermo Scientific iCAP6200 Duo Inductively coupled plasma Spectrophotometer is used for evaluation of heavy metals and toxic elements. As per available literature, the leaching of heavy metals and toxic elements namely Silver (Ag), Chromium (Cr), Cadmium (Cd), Lead (Pb) and Arsenic (As) from stabilized jarosite before and after durability study was evaluated. The range of concentrations of the above mentioned heavy metals and toxic elements, recommended by USEPA, in GGBS-lime stabilized jarosite waste blends are found to be within the permissible limits. The results are presented in Table 3.4. Figure 3.20 (a) and (b) represents the heavy metal immobilization potential of stabilized jarosite in comparison of untreated jarosite hazardous waste before and after durability respectively.

In the immobilization process during solidification/ stabilization, the heavy metals presents in hazardous waste gets converted into low soluble precipitates such as carbonates, silicates, or hydroxides and then physically encapsulated between the solids surfaces developed by formation of cementitious gels (C-S-H) (Portland cement association, 1991 [95])

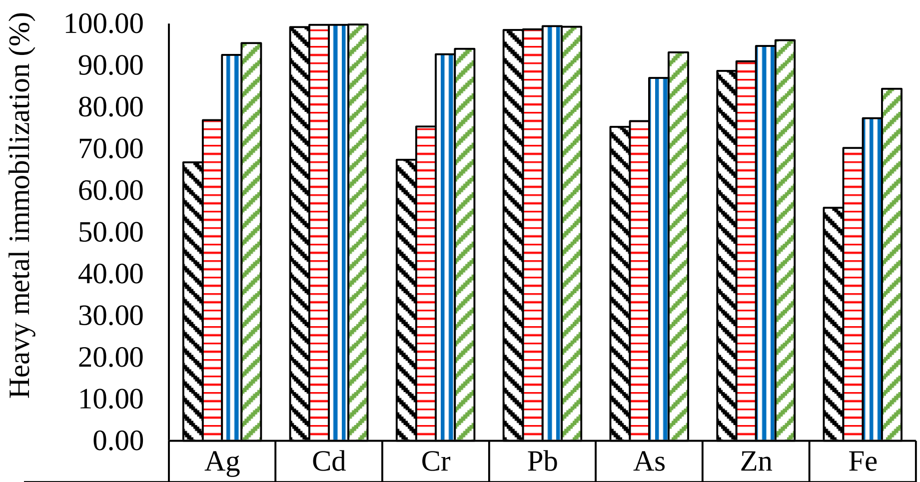
Table 3.4 Toxicity elements leachate concentration (TCLP) extract in raw jarosite waste and its stabilized composites

Heavy Metals	Untreated Jarosite	Concentration (ppm) (Before durability)				Concentration (ppm) (After durability)				US EPA Limit (ppm)
		J-10L	J-10L-10G	J-10L-20G	J-10L-30G	J-10L	J-10L-10G	J-10L-20G	J-10L-30G	
Ag	27.95	3.93	3.03	2.03	0.53	8.30	6.45	2.09	1.31	5.0
Cd	20.05	0.04	0.04	0.03	0.02	0.16	0.06	0.05	0.05	1.0
Cr	36.41	1.69	1.21	0.48	0.24	11.8	8.98	2.67	2.18	5.0
Pb	30.88	0.41	0.33	0.23	0.18	0.45	0.42	0.18	0.23	5.0
As	6.75	1.58	1.37	0.54	0.25	1.60	1.57	0.87	0.46	5.0
Zn	287.69	18.56	10.89	7.88	5.67	32.56	25.88	15.32	11.45	500
Fe	118.56	34.58	26.44	15.55	12.56	52.33	35.36	26.88	18.54	30



	Ag	Cd	Cr	Pb	As	Zn	Fe
10L	85.94	99.76	95.33	98.64	76.50	93.55	70.83
10L-10G	89.16	99.79	96.67	98.92	79.63	96.21	77.70
10L-20G	92.72	99.84	98.67	99.25	92.00	97.26	86.88
10L-30G	98.07	99.87	99.33	99.40	96.25	98.03	89.41

(a)



	Ag	Cd	Cr	Pb	As	Zn	Fe
10L	66.72	99.18	67.33	98.52	75.25	88.68	55.86
10L-10G	76.89	99.69	75.33	98.61	76.63	91.00	70.18
10L-20G	92.51	99.74	92.67	99.40	87.00	94.67	77.33
10L-30G	95.31	99.77	94.00	99.25	93.13	96.02	84.36

(b)

Figure 3.20 Heavy metal immobilization potential of stabilized jarosite, (a) Before durability; (b) After the durability

Form the results presented in Table 3.4; it is revealed that the leachate concentrations of Ag, Cd, Cr, Pb and As in untreated jarosite waste are 27.95, 20.05, 36.41, 30.88 and 6.75 ppm respectively. All these leachate concentration values in raw jarosite waste are beyond the permissible limits provided by USEPA (Table 3.4). Therefore, the jarosite waste is characterized as a hazardous waste. Similarly the leachate concentrations of Ag, Cd, Cr, Pb and As in 30% GGBS-10% lime stabilized jarosite product cured at 28 days are 0.53, 0.02, 0.24, 0.18 and 0.25 ppm and 1.31, 0.05, 2.18, 0.23, and 0.46 ppm before and after durability study respectively. Thus, the ranges of concentrations of all heavy metals and toxic elements, recommended by USEPA, in GGBS-lime stabilized jarosite waste blends are found to be within the permissible limits and hence the same are suitable for exploitation in eco-friendly applications of civil engineering.