Chapter-6

Long-term Durability of Biopolymer in Stabilization of MSWF

6.1. Introduction to the Chapter

As this thesis progresses, a pivotal exploration is reached regarding the long-term durability of biopolymer stabilization in Municipal Solid Waste Fines (MSWF).Building on the immediate stabilization effects discussed in the previous chapter, this chapter shifts the focus to the endurance and lasting effectiveness of these treatments. The significance of this research is underscored by the pressing need to develop sustainable waste management practices that extend beyond the temporary solutions often provided by traditional methods.

In this chapter, the long-term performance of MSWF stabilized with Xanthan Gum (XG) and Agar Gum (AG) is investigated. Experiments include long-term shear strength tests i.e., triaxial UU test to identify the variation of share strength, cohesion, and friction angle in long-term, microstructural analysis using Scanning Electron Microscopy (SEM). The samples are cured under constant moisture and closed conditions to simulate below-earth

crust stabilization. A comparative analysis with untreated MSWF is conducted to highlight the enhancements due to biopolymer treatments.

The findings from these experiments aim to provide a comprehensive understanding of the long-term viability of biopolymer stabilization, offering insights into sustainable waste management solutions and their practical applications in civil engineering.

6.2. Material & Method

The properties of Municipal Solid Waste (MSW) fines, sourced from a legacy waste dumpsite near the Varuna River in Sariya, Varanasi, India, have been elucidated in previous chapters. This study employs Xanthan gum (XG) and Agar gum (AG) to evaluate the long-term mechanical durability of MSW fines when treated with these biopolymers. Xanthan gum and Agar gum were selected for their superior water retention capabilities, exceptional binding properties, affordability, and availability. Both biopolymers' characteristics, including Xanthan gum's formation of a highly viscous gel upon interaction with water and Agar gum's setting into a firm gel when cooled, have been detailed in preceding chapters.

As discussed the experimental program followed ASTM standards. Samples were prepared in specific molds and extracted using a hydraulic extractor, then stored in zipped polybags at room temperature to minimize moisture loss (ref Figure 35) The samples underwent closed curing with no moisture loss for 7, 14, 28, 90, and 180 days to simulate conditions below the earth's crust, where moisture loss due to evaporation is minimal. The maximum observed moisture loss was 3%.

After curing, the samples were subjected to various tests to assess long-term mechanical durability. These included:

- Unconsolidated Undrained (UU) Triaxial Test: To evaluate the long-term effects on strength and shear properties, such as cohesion ‘c’ and angle of internal friction (ϕ).

• Scanning Electron Microscopy (SEM) Analysis: To examine microstructural changes and identify the bonding characteristics of the biopolymer-treated MSWF.

These experiments aimed to provide insights into the long-term viability of biopolymer stabilization under conditions simulating below-earth crust stabilization, with applications in sustainable waste management and civil engineering. The experimental program's flow chart is shown in Figure 35.

(a) (b)

Figure 35: Biopolymer-treated MSWF samples placed in zipped polybags for curing to ensure minimal moisture loss.

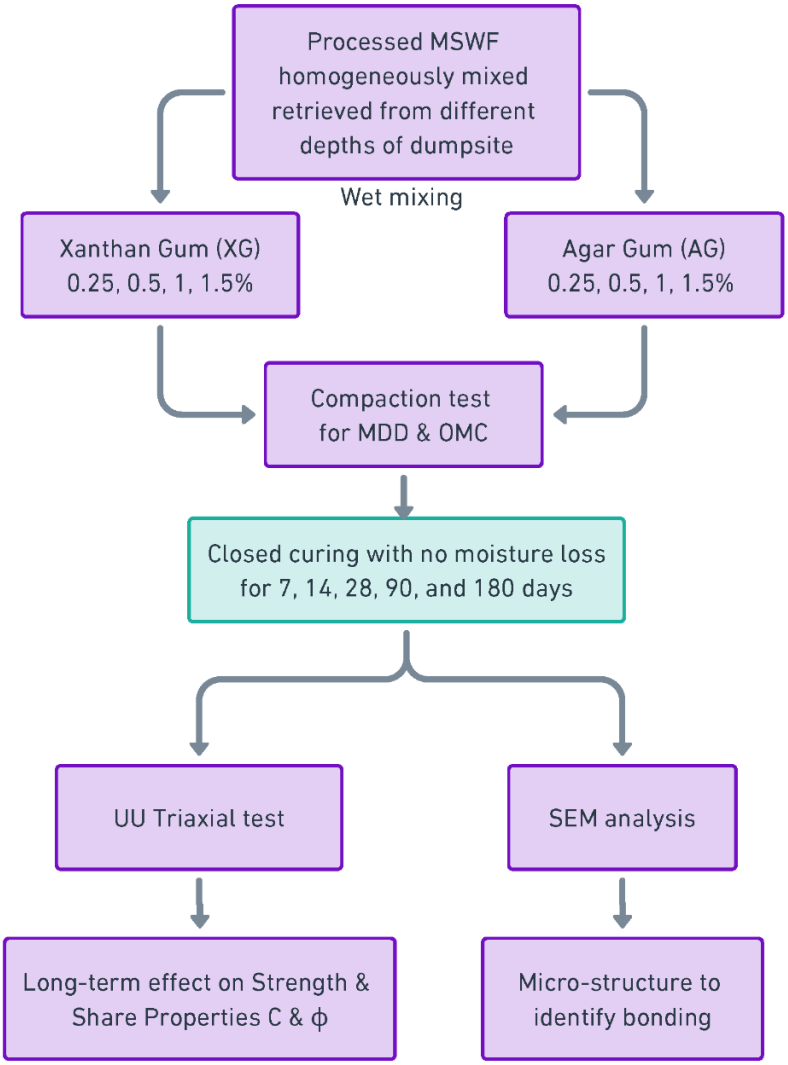


Figure 36: Flow diagram of the proposed experimental program

6.3. Results and Discussion

Previous research on biopolymers has suggested that they are promising and sustainable agents for stabilizing soil (Dehghan et al. 2019; Jang and Jia 2020; Mendonça et al. 2021). Figure 36 illustrates the mechanism of the biopolymer reaction with MSWF particles. The biopolymer forms a hydrogel when mixed with water. In this study, xanthan gum is mixed with water at room temperature, while agar gum is mixed with water at 85°C. The biopolymer gel is then mixed with MSWF, resulting in bio-clogging of the MSWF and the hydrogel surrounding the MSWF particles. Similar findings have been reported by

others(Anandha Kumar and Sujatha 2021). The biopolymer gel ultimately fills the pores in MSWF. Because MSWF particles have a negative charge around them, water-based biopolymer gel coats the particles due to a positive charge in the hydrogel (TAYTAK Başar et al. 2012). This ionic difference causes the particles to hold the hydrogel over time, a process known as bio-coating or bio-clogging. The biopolymer gel thickens naturally over time due to gradual water evaporation, leading to increased polymer concentration and enhanced cross-linking between biopolymer chains. Additionally, bio-clogging effects contribute to further densification, improving the gel's mechanical stability (Chang et al. 2020). This complex hydrogel bonding enhances the mechanical stiffness of the soil, which can be identified as the thickening, hydration, or bio-bridging stage. Later in the paper, this process will be discussed in relation to its role in improving the physical properties of the MSWF-BP mix.

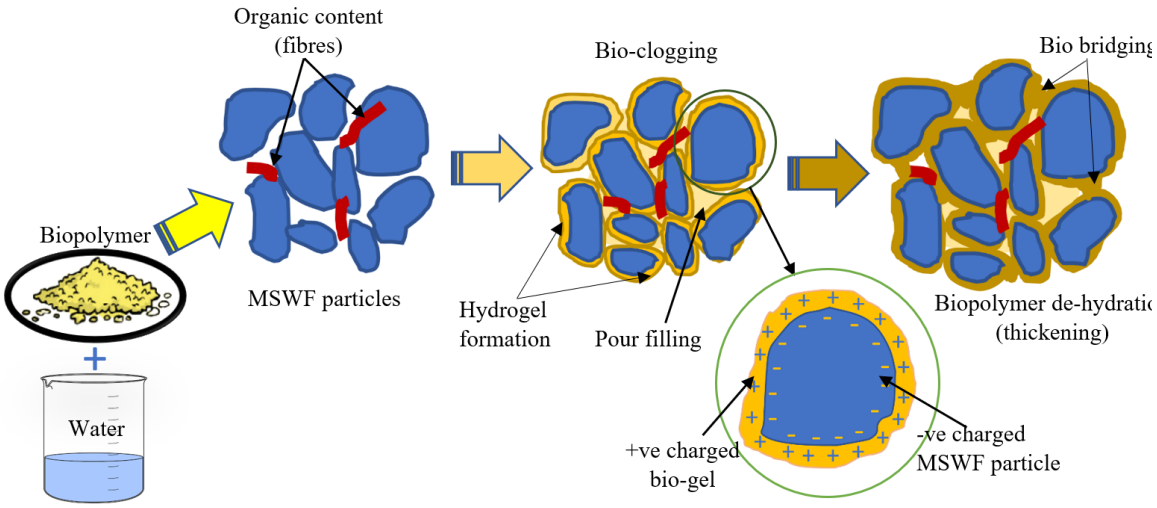
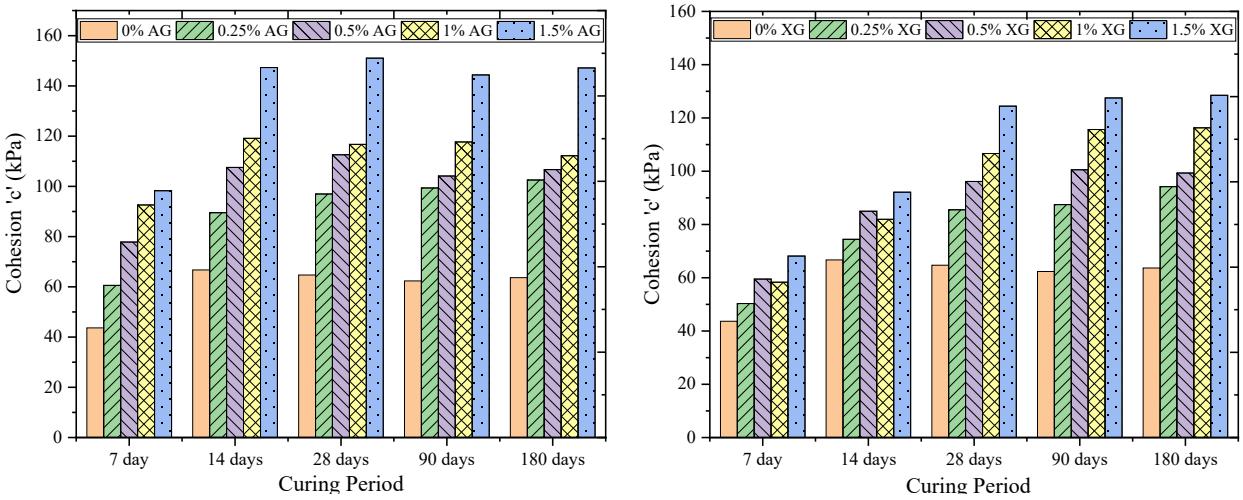


Figure 37: Mechanism of hydrogel formation, bio-clogging, bio-bridging, and hydration of biopolymer

After preparing samples with varying percentages of XG and AG, they were cured in zipped polybags to minimize moisture loss. The samples were then tested in a triaxial test apparatus following ASTM standards, and the observations are discussed in subsequent sections

6.3.1. Effect of Biopolymer on the Cohesion ‘c’ of MSWF with Curing-Time

Xanthan gum and agar gum were used as biopolymers, and their rheological properties were observed. XG formed a thick gel, while AG became a soft solid medium on cooling. Both biopolymers created a hydrogel on hydration, which plugged the pores and coated the MSWF particles, as shown in Figure 38 a,b shows that the cohesion of the MSWF and AG mix increased substantially until 14 days of curing, and then it became almost constant for the rest of the curing period. The maximum cohesion was observed for 1.5% AG (W_b/W_w) and 28 days of curing, reaching 151.1 kPa. This is because agar tends to solidify when the agar gel temperature is below 40°C, resulting in a high cohesive soil matrix. The cohesion for 7 days of curing for 1.5% AG was 98.3 kPa, and it increased by approximately 50% when the curing time increased to 14 days. A slight increase was observed till 28 days, after which a slight decrease was observed for 90 and 180 days later may be due to biopolymer degradation, moisture loss, and microcrack formation, weakening interparticle bonds (Muguda et al. 2022; Cabalar et al. 2023). The gel thinking process was observed to be very fast up to 14 days of curing, after which it slowed down.



(a) (b)

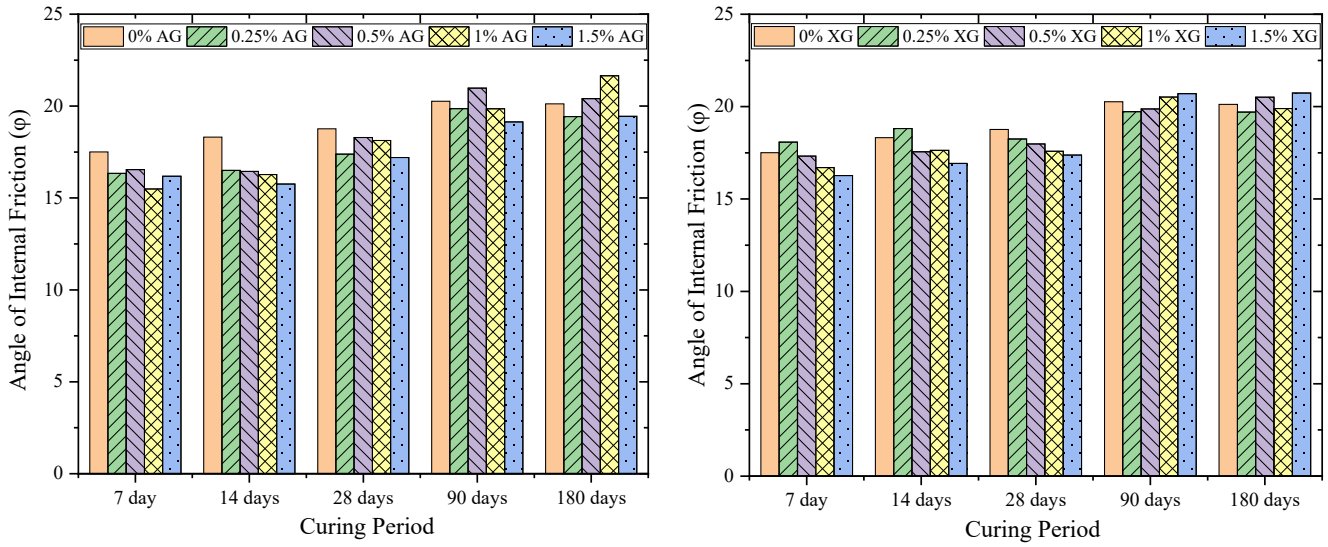
Figure 38: Variation in cohesion ‘c’ for the varying percentage of (a) AG and (b) XG

In the case of long-term curing, some amount of biodegradation in agar may occur, leading to a decrease in cohesion ‘c’. Similar findings have been reported by others (Freile-Pelegrín et al. 2007), resulting in a reduction in the cohesion of the MSWF-AG matrix. However, this degradation may be very minimal. At every curing time, the cohesion of the MSWF-AG mix increased with consistently increasing AG content. To obtain considerable cohesion in the MSWF-AG matrix, 14 days to 28 days of curing can be considered optimum.

For xanthan gum, even a small amount of gum mixed with water forms a thick gel. The XG-gel was mixed in MSWF, and the cohesion ‘c’ values were calculated for varying XG content and curing time. The values are shown in Figure 38 b where it can be seen that the cohesion ‘c’ value increases linearly till 28 days of curing time, after which the increment rate becomes very low, and the cohesion becomes almost constant. The maximum cohesion value observed was 128.5 kPa for 1.5% XG mix at 180 days of curing, and the cohesion for 28 days of curing for 1.5% XG was observed at 124.5 kPa. The increment in cohesion was only 2.4% from 28 days of curing time to 90 days of curing and only 3.3% for 180 days of curing. Hence, the optimum curing time for XG for MSWF stabilization can be taken as 28 days. The process of bio-clogging and coating is shown in Figure 37. Xanthan gum can coat the MSWF particles entirely due to its gel-like rheology when the XG thickens in pores on curing, thereby increasing the cohesiveness of the XG-MSWF matrix (Latifi et al. 2016). Similar variations in cohesion were also reported by (Ayeldeen et al. 2017; Soldo et al. 2020) when increasing biopolymer content and curing time. The average cohesion value for untreated MSWF was 60.2 kPa. The increments in cohesion were 150.8% and 113.4% for 1.5% AG and 1.5% XG for 28 days

6.3.2. Effect on the Angle of Internal Friction (Φ) with Curing Time

The angle of internal friction is a crucial soil parameter that represents the potential resistance of soil against shear failure. Typically, dense sand with angular sand particles exhibits a maximum internal friction value. However, MSWF, containing mainly silt and flaky clay particles, is a relatively cohesive soil medium. In most cases, an additive that increases soil cohesion reduces the interparticle friction of soil (Sayem et al. 2013). Figure 39 shows the plots between the angle of internal friction and curing time. It can be observed that the angle of internal friction (φ) initially decreases due to the lubricating effect of the biopolymer when the MSWF particles are coated and remain hydrated. However, as curing progresses, moisture evaporates, and the biopolymer network strengthens, improving interparticle bonding and reducing lubrication. This leads to an increase in φ over time. Conversely, at higher biopolymer contents, the lubrication effect remains dominant, causing a net reduction in internal friction. This mechanism is illustrated in Figure 37. Other researchers have also reported a similar observation (Chen et al. 2020; Adabi et al. 2022). The biopolymer hydration stage explains the increment in the angle of internal friction (φ) with time. The coating around the particle thickens with time, creating complex bonds between particles. Over time, when the sample is cured for the long term, the interparticle bonds get stronger, and it takes more effort to fail the sample in shear, resulting in an increased value of internal friction. Such finding has also been reported by (Fatehi et al. 2018) with biopolymers.



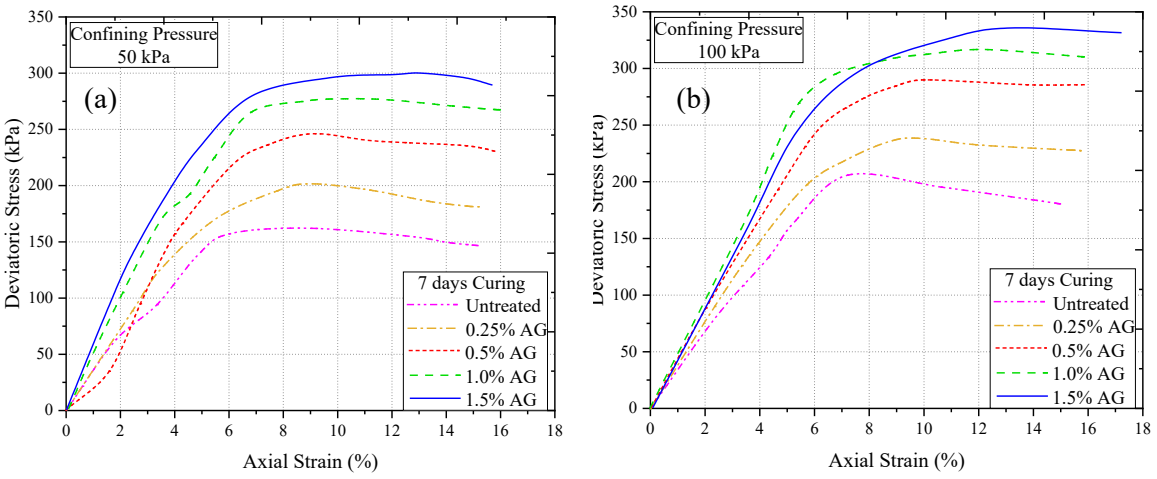
(a) (b)

Figure 39: Variation in the angle of internal friction (φ) for the varying percentage of biopolymers:

The maximum value of internal friction angle observed is 21.7° for a 1% AG-MSWF mix at six months of curing. For xanthan gum, it is 20.7° for a 1.5% XG-MSWF mix at three and six months of curing. There is a typical pattern in the angle of internal friction observed for both XG and AG. The angle of internal friction increased with increasing biopolymer content for 3 and 180 days of curing, while it decreased till 28 days of curing. This may be because the MSWF and biopolymer mix is not fully hydrated until 28 days after curing, but after 3 and 180 days, the biopolymer becomes fully hydrated and thickened, which acts as a comparatively harder particle coating. Therefore, a higher biopolymer content creates more complex bonding and retards interparticle sliding. Many researchers have examined the angle of internal friction of soil and waste material with varying curing times and biopolymer content (Smitha and Sachan 2016; Fatehi et al. 2018; Chen et al. 2020). Some have reported a decrement in the angle of internal friction with increasing biopolymer content, while others have reported an increment in the angle of internal friction with increasing biopolymer content (Chang et al. 2016; Fatehi et al. 2018; Jang and Jia 2020).

6.3.3. Stress-Strain Relation of MSWF-Biopolymer Mix

Figure 40 and Figure 41 depict the relationship between deviatoric stress and axial strain for MSWF-biopolymer mixtures. Figure 40 a-c displays the stress-strain graphs for samples stabilized with AG and cured for 7 days, while Figure 40 d-f shows the same for samples stabilized with XG and cured for 14 days. It can be observed that both untreated and treated samples exhibit ductile behaviour, with yielding occurring at 5-8% axial strain. The ultimate strain is recorded between 8-14% axial strain and increases with increasing biopolymer content. Once the ultimate stress is reached, all MSWF samples exhibit strain-softening behavior. This behavior may be attributed to the biopolymer's tendency to increase soil cohesion and form hydrogel bonds. The sample's ductility increases as cohesion increases, and the ultimate deviator stress increases as interparticle bonding increases. This observation is consistent with Khatami and O'Kelly's findings (Khatami and O'Kelly 2013).



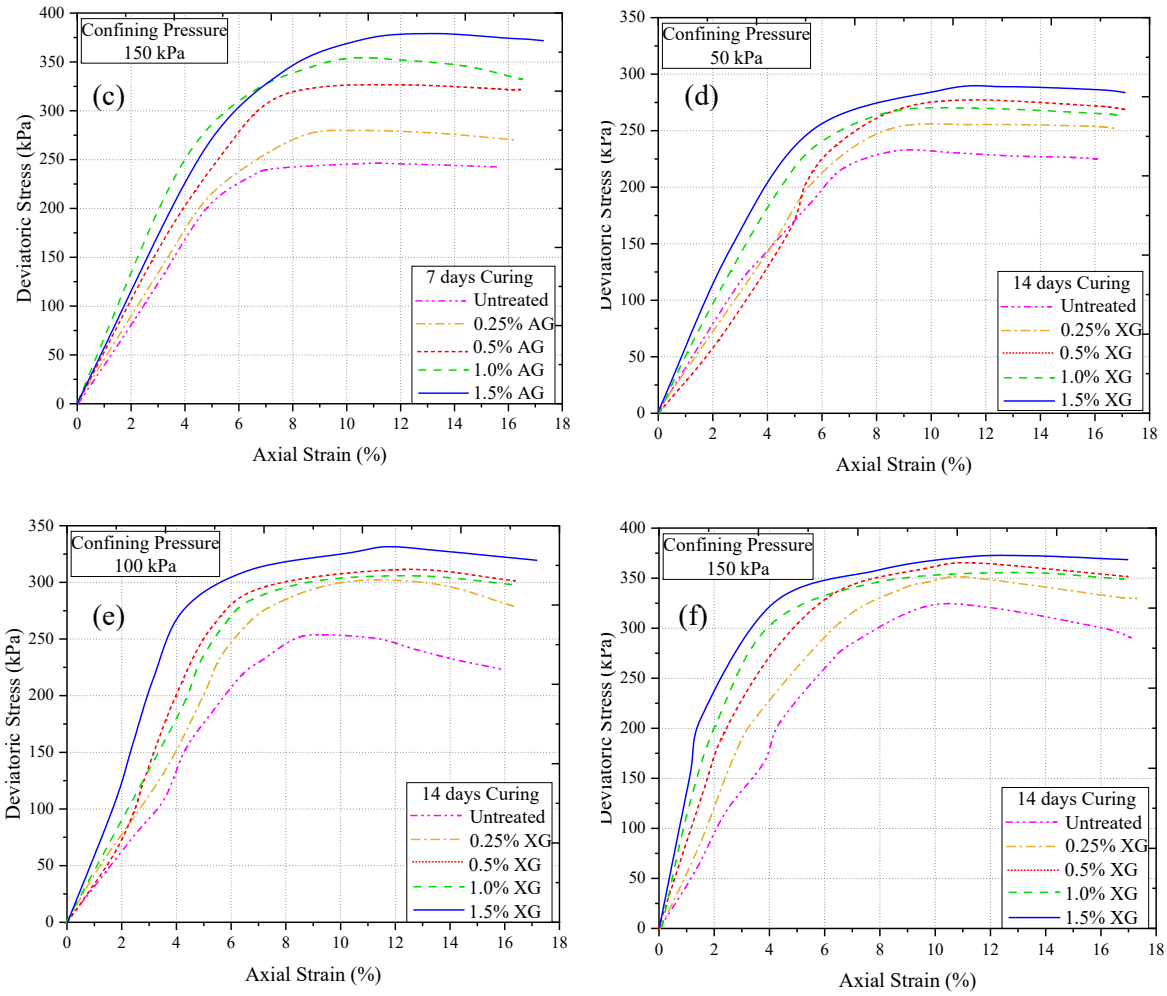


Figure 40: Plots b/w deviatoric stress and axial strain for 7 days and 14 days cured MSWF samples treated with AG and

Figure 41 illustrates the stress-strain behaviour of MSWF samples stabilized with AG and XG and cured for 180 days. It is evident that the ultimate deviatoric stress and strain have significantly increased. For 1.5% XG and AG, the corresponding ultimate deviatoric stress occurs between 16-20% axial strain. However, the graphs in Figure 41 d-f exhibit slightly more skewness than those in Figure 41 a-c due to XG's higher yielding strain compared to AG after 180 days of curing.

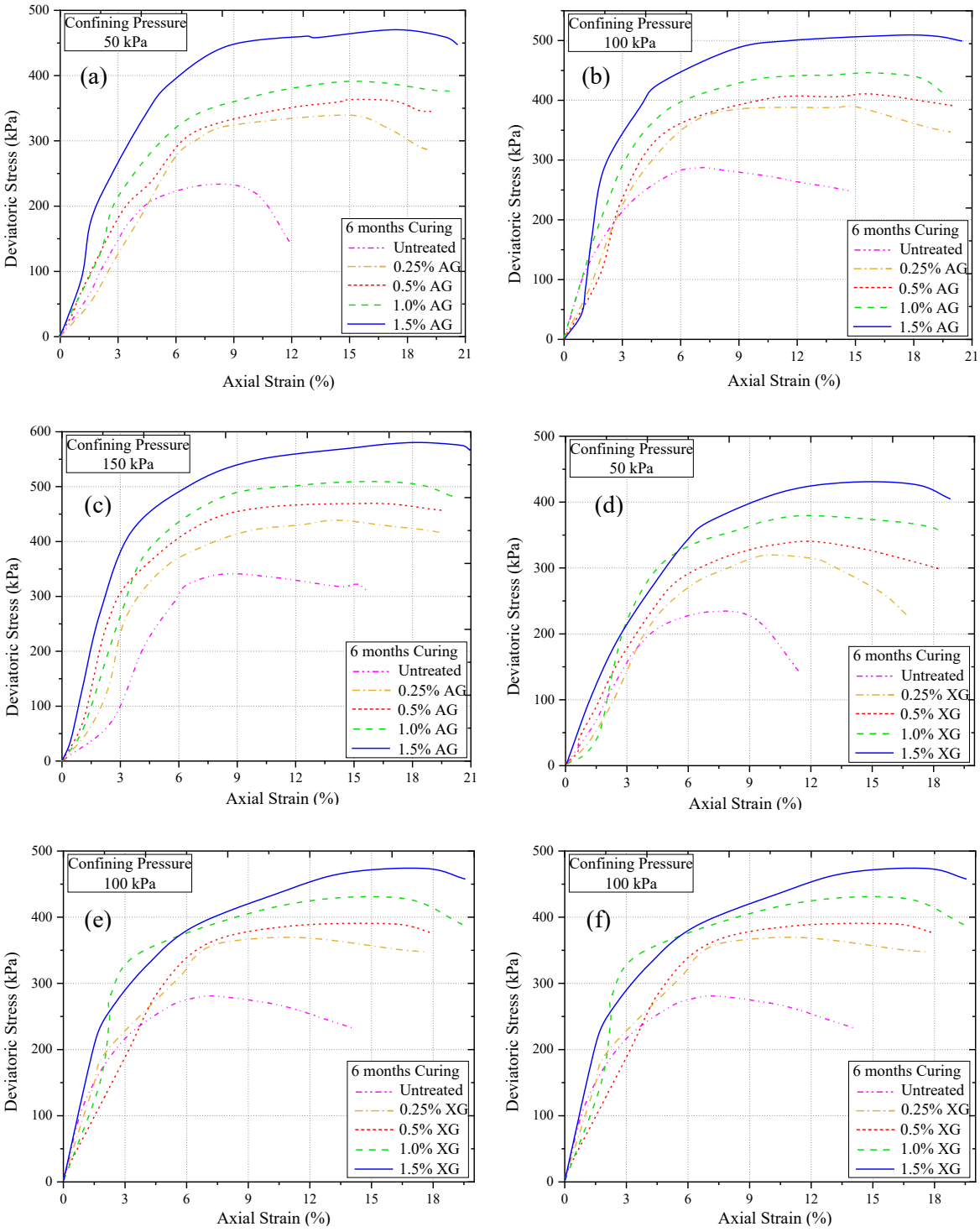


Figure 41: Plots between deviatoric stress and axial strain for 180 days cured MSWF treated with AG and XG

6.3.4. Effect of Curing Period and Biopolymer Content on Shear Strength

The shear strength of the MSWF-biopolymer mix was calculated using the respective c and φ parameters, and the plots are presented in Figure 42. In the case of agar gum, the shear

strength of the MSWF-AG mix increased rapidly until 14 days of curing, after which the rate of increment reduced. The shear strength became almost constant after 90 days of curing due to biopolymer hydration and thickening in constant moisture conditions. Similar findings have been reported by other researchers (Smitha and Sachan 2016; Rashid et al. 2019). Analysis of the 'c' and 'φ' parameters showed that changes in these parameters were significant for the first 14 days of curing and then became modest. When agar gum is mixed in the soil, it solidifies to 40°C and combines with MSWF particles during solidification, but it does not entirely hydrate within the soil. As time passes and the biopolymer dehydrates, the biopolymer bonds between particles thicken, increasing the sharing resistance of the MSWF-AG matrix (Chen et al. 2015, 2019). The hydration of agar gum particles is highly active until 14 days, during which agar creates more complex bonds with soil particles, after which it settles down, and the sample reaches its maximum strength. Although a relatively low strength increment was observed after 14 days, this can be understood because the hydration of biopolymer and soil particles and dehydration of interconnecting bonds are continuous processes. As the interconnecting bonds thicken, they create more complex interparticle bonding (Chang et al. 2015). The maximum shear strength observed in AG's case is 329.2 kPa, recorded at 1.5% AG-MSWF mix on 180 days of curing.

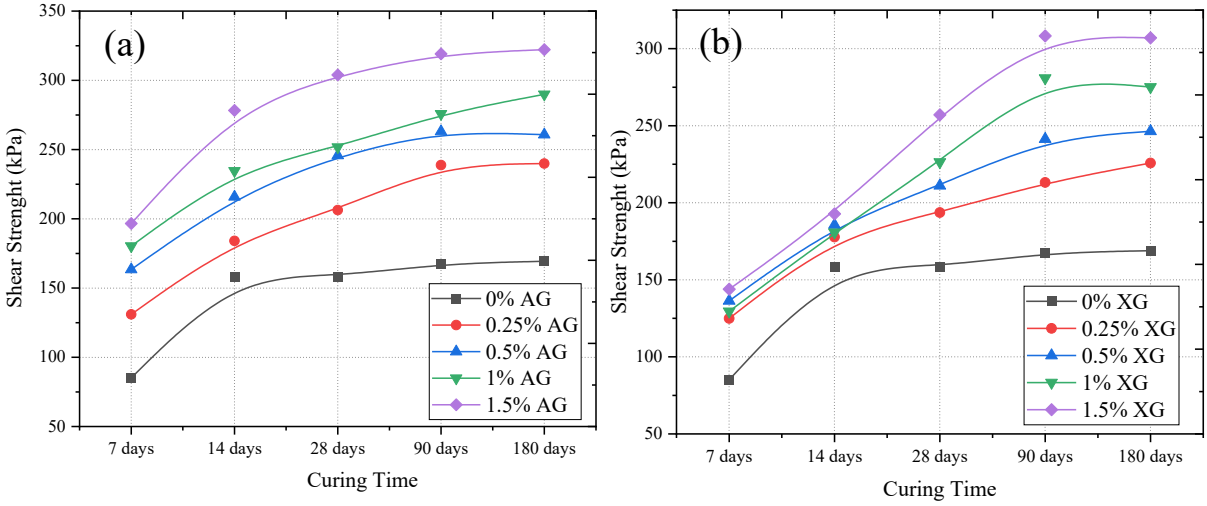


Figure 42: Variation of shear strength with curing time (a) Agar Gum (b) Xanthan Gum.

Active hydration and bio-clogging were observed in samples stabilized with xanthan gum (XG) for up to 90 days of curing. After this period, XG stabilized in the mix of municipal solid waste fly ash (MSWF) and XG. A significant increase in shear strength was observed for up to 90 days of curing in MSWF-XG mixed samples. Comparing the rheology of AG and XG, it was observed that XG remained in gel form for a longer period in the MSWF matrix compared to AG. It slowly congealed and created bonds with particles. At 90 days of curing, the maximum shear strength of the MSWF-XG mix was 308.3 kPa, while at six months of curing, it was 310.9 kPa. The fibres present in MSWF also played a role in altering the shear strength parameters of MSWF (Figure 37) (Rawat and Mohanty 2021). Due to the adhesive nature of clay particles and biopolymer, fibers entered the soil matrix to create bonds with MSWF particles and add strength to MSWF samples. Comparing the maximum shear strength, the strength gain from 14 days to 180 days was only 18.3% in the MSWF-AG mix, while up to 61.3% was observed in the MSWF-XG mix. The shear strength increased by 264.9% and 286.4% for MSWF-XG and MSWF-AG mix, respectively, compared to the untreated sample (1.5% biopolymer and 180 days curing). The shear strength increased with increasing biopolymer content in MSWF, which is

common in AG and XG. This trend was observed elsewhere (Khatami and O'Kelly 2013; Chang et al. 2016; Cabalar et al. 2017).

6.3.5. SEM Analysis of MSWF Stabilized with Biopolymer

SEM analysis at 5000x magnification was used to determine the morphology of treated and untreated samples. The FESEM machine Quanta 220F (FEI Netherlands) was used for this analysis. SEM images of typical samples are presented in Figure 43 a shows the SEM image of the untreated MSWF sample, Figure 43 b & c shows the 14 days cured sample treated with 1.5% AG & XG, and Figure 43 d-e shows the SEM images of the 180 days cured sample treated with 1.5% AG and XG, respectively. In the SEM image of the untreated sample, a significant amount of pores and voids are visible, and the individual MSWF particle is clearly visible in Figure 43 b-c, the pores are partially filled due to the hydrogel created by the biopolymer. After 14 days of curing, the hydrogel partially filled the pores in the MSWF matrix, but the biopolymer's bio-clogging and thickening were still ongoing. By visual analysis of Figure 43 d-e, it is apparent that the biopolymer gel excessively filled the pores in the sample, leading to the interconnecting particles of MSWF. Comparing Figure 43 d-e, the AG-stabilized samples show a denser profile than the XG-stabilized samples, which aligns with earlier identified trends. In the present study, AG-stabilized samples showed better mechanical performance than XG. Hence, it can be said that AG comparatively created more hydrogel, and the pore filling was more in the case of AG.

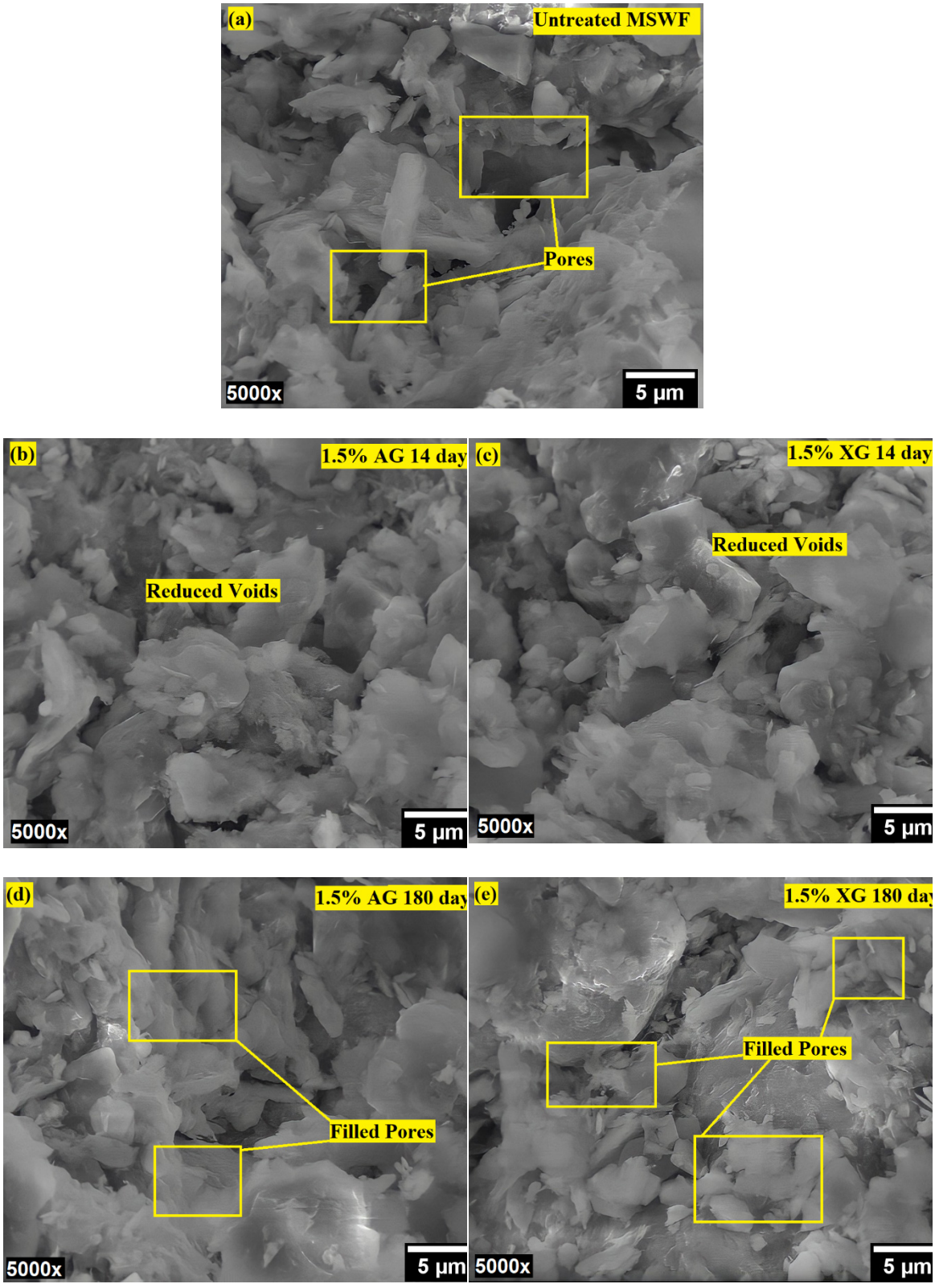


Figure 43: SEM image of untreated and treated MSWF samples at 5000x magnification.

6.4. Key Takeaways and Way forward

- AG-treated MSWF showed near-constant cohesiveness after 14 days, while XG-treated MSWF peaked at 28 days, indicating different stabilization dynamics.
- The internal friction angle decreased with increased biopolymer content up to 28 days, then increased at 90 and 180 days, indicating stronger long-term interparticle bonds.
- XG-treated samples displayed higher ductility than AG after 180 days. AG's shear strength rapidly increased until day 14, while XG saw significant strength gains primarily during the first 90 days.
- Curing filled MSWF matrix pores over time, resulting in a denser biopolymer-soil composite structure.
- Biopolymer concentrations could potentially increase strenght by over 450%.

The next chapter, **Civil Engineering Applications** will apply the durability findings to practical scenarios, assessing the use of both treated and untreated MSWF in construction projects like embankments and subgrades.