

Characterization of Cassava Starch Films Strengthened with Glass Particulate Composite

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Abstract: Characterization of cassava starch films strengthened with glass particulate composite was investigated in this study. Glass particulate starch composites was produced using solution casting process with weight percentage (wt.%) of the reinforcement phase ranging from 0% to 20% at 5wt.% interval. The mechanical and physical properties were determined out by standard methods. Themicrostructural analysis of the produced composites was carried out using scanning electron microscope (SEM). The density of the composites increased as the reinforcement content increases, the water absorption decreased as the reinforcement content increases. The SEM analysis showed good adhesion between the starch matrix and the reinforcement, the hardness of the bio-composites improved generally over the unreinforced starch and the biodegradability of the thermoplastic polymer (starch) degrades faster when compared with the bio-composites. The development of the bio-composite will contribute to knowledge; helps convert waste to wealth and reduce environmental pollution. The bio-composite produced has light weight and can be used in food packaging applications.

Keywords: Starch, Glycerol, Glass particles, Solution casting, Physical properties

I. Introduction

In the last few years there has been a great interest in the development of green technologies around the world for products that have lower environmental impact. Green chemistry, as a whole, involves the development of chemical processes and products that generate a cleaner, healthier and sustainable environment (Silva *et al.*, 2012). Thus, synthetic plastic materials have received much attention because of their non-biodegradability and non-renewable sources (Zhong *et al.*, 2012; Meneguín *et al.*, 2017; Chaichi *et al.*, 2017).

One solution found to improve the environmental impact of synthetic plastics was the development of biomaterials from renewable polymers that can substitute synthetic materials. Starches are polymers with a high potential to produce flexible films and are inexpensive, biodegradable and highly available from renewable sources. However, the primary challenge is to substitute conventional packages while maintaining the same efficacy, quality and shelf-life. These results can be obtained through the control of mechanical properties and permeability (Henrique *et al.*, 2007; Seligra *et al.*, 2016; Qazanfarzadeh and Kadivar, 2016; Montero *et al.*, 2017).

Starch is one of the most used materials for producing biodegradable plastics, being naturally renewable, cheap and plentiful (Nattakanet *et al.*, 2012). However, films formed from starch are brittle and difficult to handle, so different plasticizers are normally added to the film-forming solution before doing the casting and drying procedures to obtain thermoplastic starch (TPS) (Qiu *et al.*, 2013). Glycerol, which is becoming nowadays a waste product generated by the biofuel industry, gives the best results in decreasing the friction between starch molecules (Zainuddina *et al.*, 2013).

To improve the characteristics of starch-based film, many researchers have shown an interest in using fillers and fibers, particularly cellulose fibers, as a reinforcement in TPS matrices in the preparation of green composites; the cellulose fibers are obtained from different sources such as kenaf fiber (Zainuddina *et al.*, 2013), jute (Nattakanet *et al.*, 2012), ramie (Yongshang *et al.*, 2006), recycled paper cellulose fibers and lignocellulose-based fibers (Avérous and Digabel, 2006), cellulosic fibers from *Eucalyptus urograndis* pulp (Curvelo *et al.*, 2001) and sisal-coir fibers (Arya *et al.*, 2017). The major attractions of these green composites are their good tensile properties, which are attributed to the chemical compatibility between starch and cellulose; their high resistance to water because of the hydrophobic character of fibers; and, also, their full degradability and sustainability (Avérous *et al.*, 2001).

Yonas *et al.*, (2022) reported that the use of waste glass particles as reinforcement in Al2024 alloy matrix improves the composite hardness, tensile strength and corrosion resistance.

Based on literatures, there is lack of adequate knowledge on the use of glass particles as reinforcement in polymer based matrix composites, as most researches are focussed on the use of glass fibre reinforced polymer composites.

In this present study, An investigation was carried out on the use of glass particles as reinforcement in cassava starch composite in other to produce materials with improved properties. The morphological, physical and mechanical behaviour of the prepared green composites were determined by standard methods equipment. The developed bio-composite can be used in food packaging applications.

II. Materials And Method

Materials

The materials that were utilized for this study are native cassava starch and glass particles. Native cassava starch was sourced from Lokoja International Market, Kogi State, Nigeria while glass bottles were sourced from local retailers shop. The commercial grade glycerol was procured from Fodayomi Chemical Company, Lokoja.

Charge preparation

The charge materials were cassava starch and glass particles. The glass bottles were washed and dried for two days; After drying they were crushed to smaller size using a jaw crusher machine. The crushed particles were further pulverized to powder form of size <10µm.

Synthesis of the composite

The cassava starch films were prepared using hand lay-up casting procedure. the starch solution was prepared in a conical flask and mixed with 20% of glycerol (Yongshan*et al.*, 2006). The mixture was heated to 100 °C and shaken to ensure homogenization. The glass particles were added to the gelatinized solution and the composite was poured into the wooden mold (50 x 30 x20) cm to cure. The first cast was for the control sample (unreinforced starch) while the subsequent cast were for 5wt%, 10wt%, 15wt% and 20wt respectively. Finally the castings were removed and machined into the desire test samples.

Physical Properties

Water absorption test

Water absorption test was carried out according to ASTM D570 standard (Rabiu and Ramalan, 2020) by recording the mass of dry specimen using an electronic weighing balance. The weighed samples were immersed in distilled water for 24 hours, the samples were removed, dried and then weighed again. The percentage increase in weight is the water absorption of the bio-composite.

$$\text{Water absorption} = \frac{m_2 - m_1}{m_1} \times 100\% \dots\dots\dots 1$$

m_2 = final mass of sample

m_1 = initial mass of sample

Density measurement

The density of the composites were carried out using mass and volume relationship based on Archimedes principle. Samples were weighed using an electronic weighing device and then immersed in a calibrated measuring cylinder containing water. The displaced volume is the volume of the sample. The respective densities are calculated (Mohsin*et al.*, 2019).

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} \dots\dots\dots 2$$

Biodegradability test

The biodegradability test of the composites were carried out according to ASTM C1327 standards (Sathishkumar *et al.*, 2018). The oven dried samples were buried in the soil at a depth of 25cm for 60 days. The samples were removed after 60 days, cleaned and oven dried, and then weighed by applying weight loss calculations (Patel and Sen, 2011).

$$\text{Weight loss} = \frac{w_i - w_f}{w_i} \times 100 \dots\dots\dots 3$$

W_i = initial weight of the sample

W_f = final weight of the sample

Characterization Techniques

X- ray fluorescence (XRF)

XRF was carried out using Cu-Zn method, the 20wt% bio-composite was polished and subjected to xrf analysis. The oxide compositions were identified through XRF analysis

X-ray diffraction analysis

The XRD diffraction pattern was recorded by an X-ray diffractometer (EMPYREAN). The machine was operated at 45 KV voltage and 40 mA using Cu $K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) in the angle range of 20-80°. The specimen was ground and polished before the XRD test.

Scanning electron microscopy

Detailed microstructural features were performed using scanning electron microscope. The unreinforced starch (0 wt%) and the 20wt% specimen were observed under SEM and micrographs were taken by the scanning electron microscopes (Model: FOV USM-767). The images were taken at various magnifications.

Mechanical Test

Hardness

Vickers Hardness test was carried out using Finlab Vickers Hardness Tester, model MVI-Pc, NO. 07/2012-1329. The samples were subjected to a direct load of 0.3kgf at a minimum and maximum load of 20HV and 150HV respectively through the longitudinal section according to ASTM D2240 (2015). Three indentation points were recorded and the average was computed.

III. Results and Discussion

Physical Properties results

Water absorption

The water absorption test results is presented in Figure 1. From the Figure 1, it can be seen that the presence of glass particles caused a decrease in water absorption of the developed bio-composite. For the steady state water absorption, the highest water absorption was observed in the 0wt% (starch composite) at 64.46% while the least was observed in the 20wt% composite at 44.18%. The decrease in water absorption may be attributed to the presence of hard glass particles in the composite(Rabiu and Ramalan, 2019).

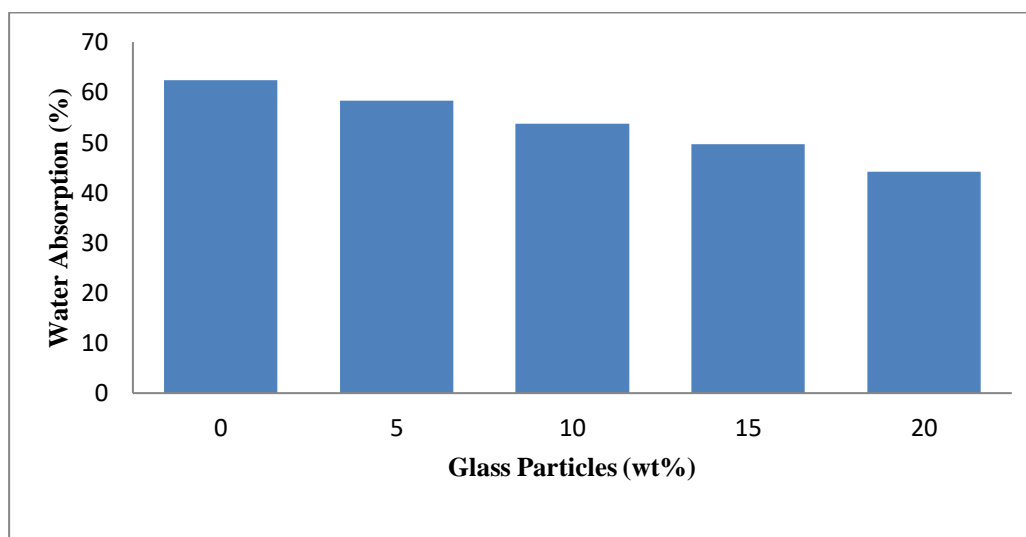


Figure 1: Variations of water absorption with glass particles

Biodegradability

Figure 2 shows the biodegradability result. It can be observed that the thermoplastic polymer (starch) degrades faster when compared with the bio-composites. The 0 wt% (unreinforced starch) had the highest degradability value at 52.39% and the least degradability value was observed in the 20wt% composite at 63.42%. Weight loss increased as % reinforcment increases; most micro organisms depends on temperature, available oxygen and matrix stability in the biodegradation process (Patel and Sen, 2011)

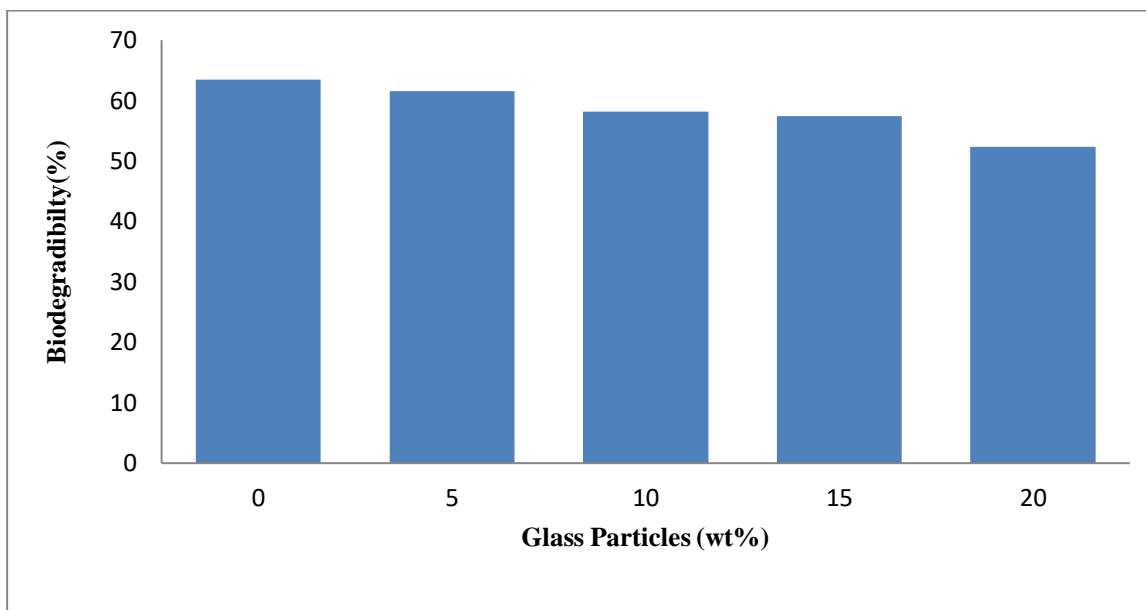


Figure 2: Variations of biodegradability with glass particles

Density

Figure 3 shows the density values for the starch and composite material at different particle content. Figure 3 illustrates an increase in density value as the reinforcement content increased. The highest value was recorded as 2.2 kg/m³ in the 20wt% composite and the least value was recorded for the unreinforced starch at 1.8 kg/m³ . The trend can be attributed to the particulate been heavier and denser than the matrix material (Seligra *et al.*, 2016). Therefore an increase in glass content particles increased the overall density of the composite material.

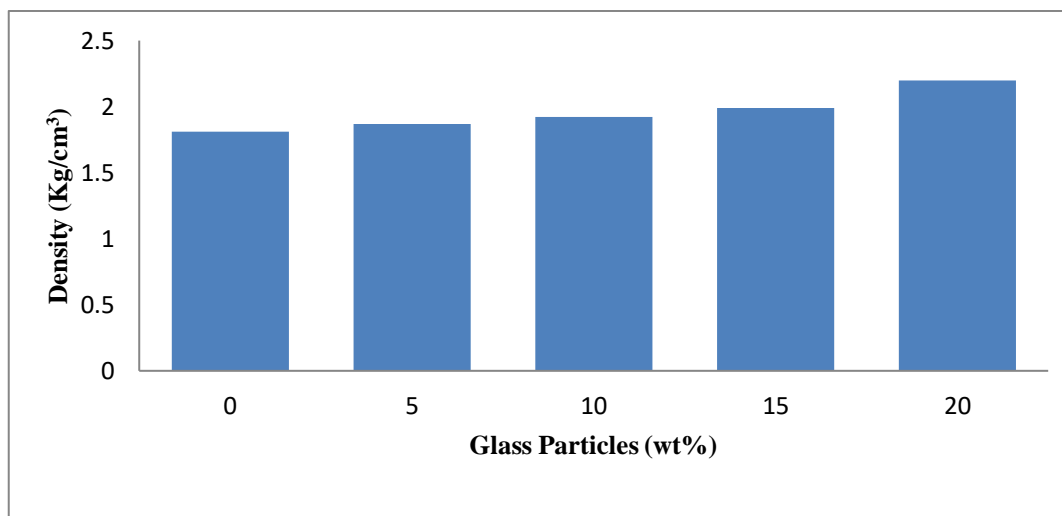


Figure 3: Variations of density with glass particles

Mechanical Property Result

Hardness test analysis

The results of the hardness test is presented in the Figure 4. Figure 4 shows that the hardness value of the composite increases as the content of the glass particles increased with the highest value recorded at 43.23 HV for the 20wt% composite while the unreinforced starch the least with 35.21 HV. This behaviour can be attributed to the glass particles been a harder material compared to the base matrix material. Increasing the concentration of the particulate result in higher indentation resistance and subsequently higher hardness value (Abbas *et al.*, 2020).

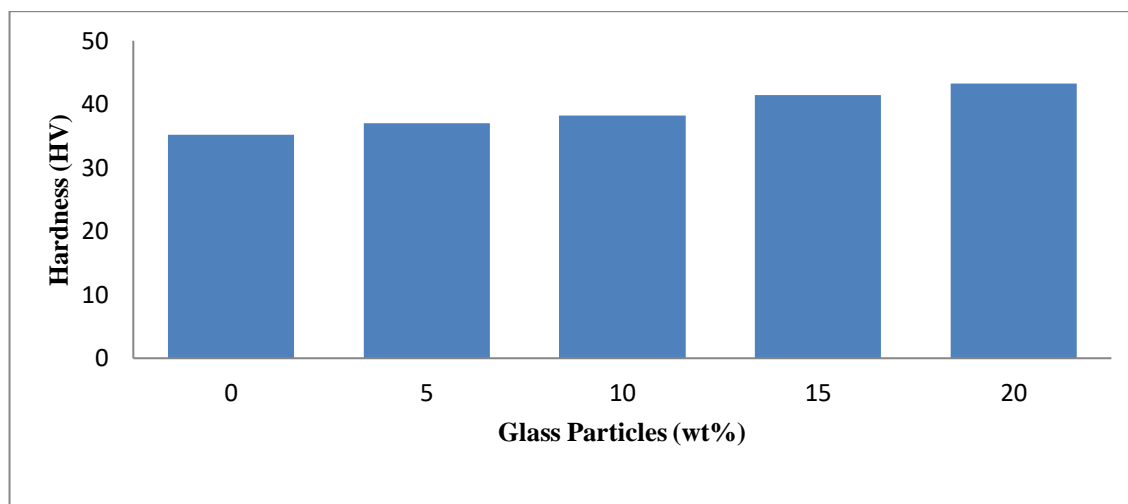


Figure 4: Variations of hardness with glass particles

Characterization Analysis

X-ray fluorescence (Xrf)

The X-ray fluorescence results is shown in Table 1.

Table 1: XRF results of 20wt% bio-composite

Oxide composition	Composition(%)
Ash (Carbon)	6
Moisture (H ₂ O)	12
Fat (CH ₃ (CH ₂) _n COOH)	3.8
Protein (RCH (NH ₂) COOH)	0.2
Carbohydrates (CH ₃ (CH ₂) _n COOH)	78

From Table 1, the composite is majorly carbohydrate at 78%, followed by moisture at 12%, then ash at 6%, while protein is the least with a value of 0.2%. The results confirms that cassava powder has a higher carbohydrate concentration. This shows the starchy composition of the material is utilized in this study (Zainuddina*et al.*, 2013).

X-ray diffraction

Figure 5 shows the unreinforced starch as well as bio-composites with diffraction peaks at 2θ . It can be observed that as weight percent reinforcement increases the peaks pattern initially increased and subsequently decreased. The addition of glass particles caused alterations in the in the diffraction patterns of bio-composites. The crystalline peaks occurring at around 2θ of 20° were due to the precipitation of helical inclusion complexes formed among amylose, plasticisers, and lipids at the time of extrusion (Sun *et al.*, 2014). The peak shifts and the decrease in intensity indicate that the system was partially exfoliated (Katerinopoulou*et al.*, 2014).

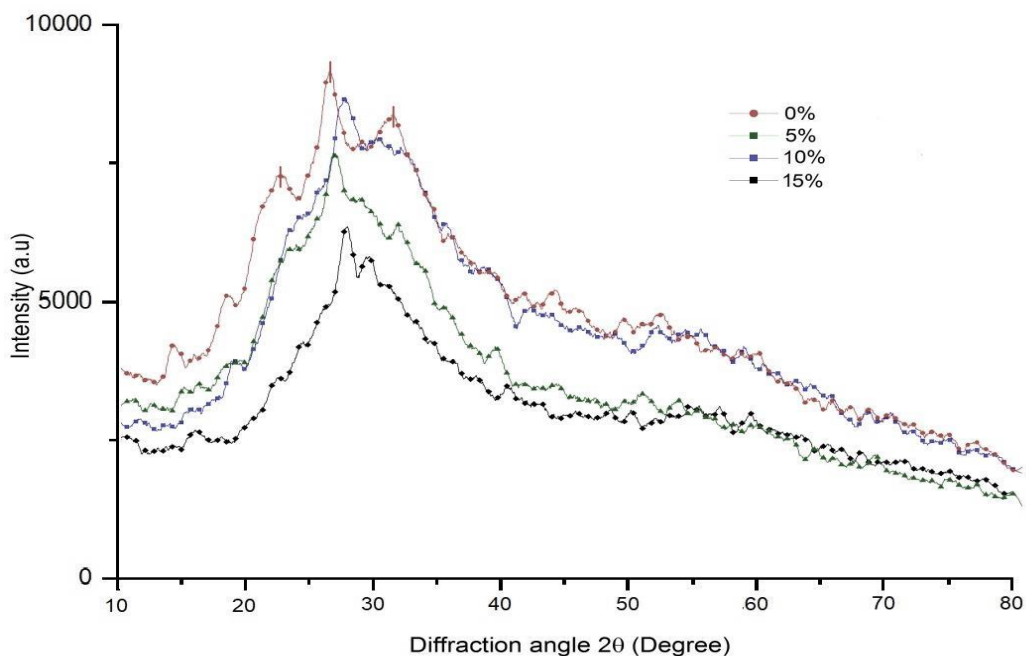


Figure 5: XRD results of starch/ glass particles bio-composites

Scanning electron microscopy (SEM)

Plate I and Plate II shows the surface SEM of unreinforced starch and 20wt% composites respectively. It can be observed that the microstructures shows a uniform and smooth surfaces with a homogeneous mixture likewise good adhesion of the glass particles in the thermoplastic starch composites. The white patterns are the starch rich phase and the dark region is the glass particles phase. Similar observations have been made on starch based composites (AverousandBoquillon,2006)

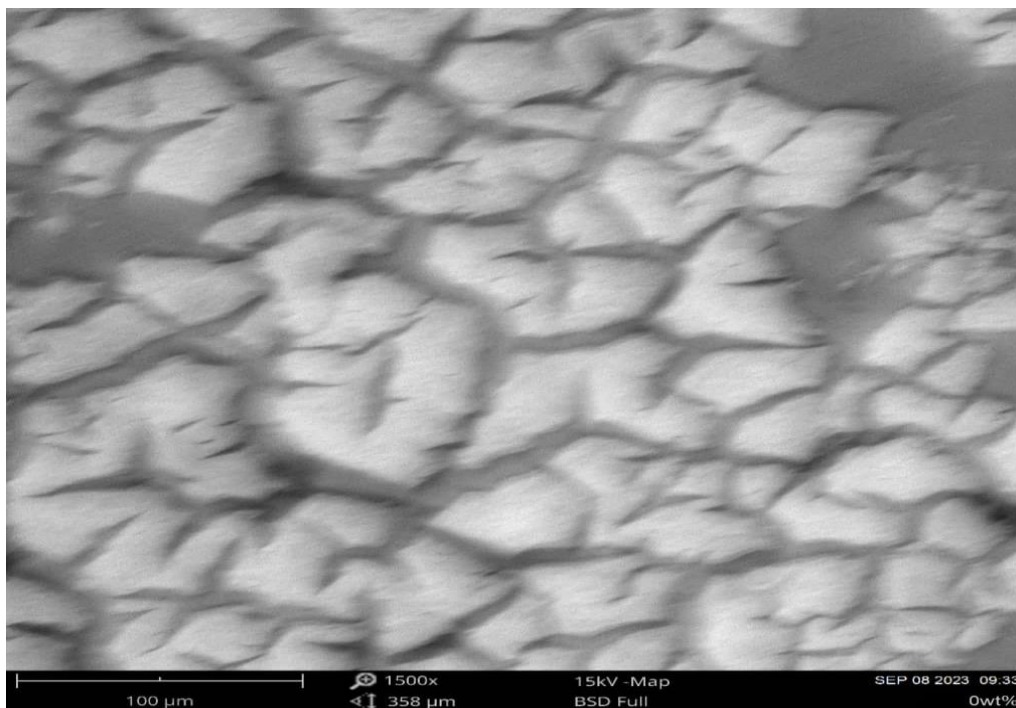


Plate I: SEM of unreinforced starch

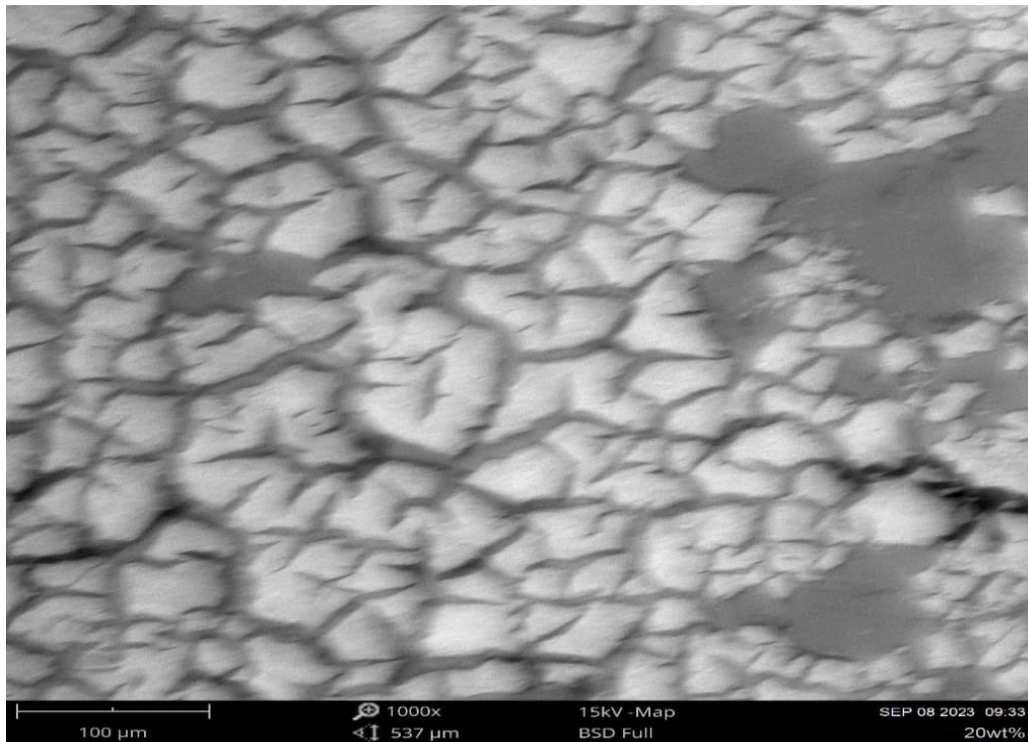


Plate II: SEM of 20wt% composite

IV. Conclusion

The following conclusions have been drawn

- I. The starch based bio-composites can be prepared by solution casting process
- II. The bio-composites were developed with uniform dispersion in the castings
- III. The density of the composites increased as the reinforcement content increases
- IV. The water absorption decreased as the reinforcement content increases
- V. The SEM analysis showed good adhesion between the starch matrix and the reinforcement
- VI. The hardness bio-composites improved generally over the unreinforced starch
- VII. The biodegradability of the thermoplastic polymer (starch) degrades faster when compared with the composites

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