

EXPERIMENTAL ANALYSIS TAPIOCA STARCH REINFORCED ALUMINA NANOPARTICLE (Al_2O_3) COMPOSITE

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ABSTRACT

The starch-alumina nanoparticle composite components combined together whereas tapioca starch as a matrix and alumina nanoparticle (Al_2O_3) as a filler. By increasing the weight percentage of alumina nanoparticle (Al_2O_3) into starch composite, the tensile strength of the composite to frack increase linearly with the increasing of the alumina nanoparticle (Al_2O_3). The exact reason for this variation can be attributed to the fact that alumina particles play like stress concentrators, and this role is promoted as particles lead to be agglomerated. An agglomeration of filler particles play an important role in creation of stress concentration that effect the composite cracks. The brittle result from the graph applied and the displacement (elongation) before frack decreasing with the increasing alumina nanoparticle (Al_2O_3). Lateral gage middle, angled gage middle, lateral at grip/tab top are tensile type failure codes/typical modes. Scanning Electron Microscopy showed good adhesion between tapioca starch and alumina nanoparticle (Al_2O_3). The adhesion properties between tapioca starch and alumina nanoparticle (Al_2O_3) strong relate to the effects of increasing tensile strength and decreasing in elongation. Three different cracks regions including a flat featureless mirror zone surrounding the crack initiation point, a transition zone, in which the surface roughness steadily increases, and a final propagation zone with conical marks.

Keywords: Tapioca starch, alumina nanoparticle (Al_2O_3), tensile strength, scanning electron microscopy

1. Introduction

In the past few decades, environmental concerns about an increase in non-degradable plastic waste have generated significant interest in biopolymers from renewable natural sources. The appeal of the protein-based biopolymer is that they can serve as an alternative to conventional petroleum-based plastics because of their nontoxic, large-scale availability, low cost, biodegradable and environmentally friendly properties (Sudsiri et al 2011). There is an increase in public awareness of environmental issues, particularly surrounding the disposal of packaging. Increased use of synthetic packaging films had led to serious ecological problems due to their non-biodegradability. The use of biopolymers can be an important tool in environmentally friendly management because of the large amount of polymers used in many applications.

Starch is one of the most commonly used raw materials to prepare biodegradable film because it an agro-sourced polymer has received much attention recently due to its strong advantages such as low cost, wide availability, and total compostability without toxic residues. However, despite considerable commercial products being available, the fundamental properties (mechanical properties, moisture sensitivity) of plasticised starch-based materials have to be enhanced to enable such materials to be truly competitive with traditional petroleum-based plastics over a wider range of applications. Regarding this, one of the most promising technical advances has been the development of nano-biocomposites, namely dispersion of nano-sized filler into a starch biopolymer matrix (Fengwei et al 2013).

However, starch-based materials are known to have limitations such as poor processability and properties (e.g. weak mechanical properties, poor long-term stability, and high water sensitivity). Formulation development and understanding of starch thermal (Liu et al 2009) and rheological (Xie et al 2012) properties could be the keys to solve these critical problems. Further, various starch-based blends and biocomposites have been developed, showing improved performance (Avérous 2004). Recently, along with the exponential momentum of the development in polymer nanocomposites (Alexandre and Dubois, 2000), much attention has been focused on the use of nano-sized fillers in improving the performance of and adding new functionalities to starch-based materials.

Various nanofillers have been examined with plasticized starch, including phyllosilicates, polysaccharide nanofillers, carbonaceous nanofillers, and many more. They have different geometry (size and shape) and surface chemistry. Regarding their shapes, three distinct types of nanofillers can be observed such as nanoparticles, nanotubes, and nanolayers (Kumar et al 2009). Nevertheless, the term “nanoparticles” is also frequently used in interesting to both fundamental research and industrial applications.

Alumina based fibers are attractive because of their excellent properties at high temperature and oxidizing atmosphere environment. Mullite and corundum are the main phases of the alumina based fiber. Mullite ($3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$) is the only stable intermediate phase in the $\text{Al}_2\text{O}_3\text{--SiO}_2$ system at atmospheric pressure (Duval et al 2008). The creep resistance of mullite is much higher than alumina. So the existence of mullite phase improved the creep resistance of fiber significantly. The fiber is mainly used in two areas: as reinforcement of metals, ceramics or resins in the form of continuous fibers (Boccaccini et al 2005) and as high temperature insulating material in the form of mats, blankets. (Zhang et al 2008) Particularly it is irreplaceable as the reinforcement in high temperature ceramic matrix composites, which has been widely used as thermal insulation material of aerospace craft and as advanced engine components in the civil automotive industry. However, it is also facing challenges due to their low strength compared to carbon fibers or carbide/nitride fibers.

Interfacial strength between the polymer and inorganic fillers is an important factor in the composite properties. Strong polymer filler interface bond plays a decisive role in achieving good mechanical properties of the composites (Mohanty et al 2001). To enhance the polymer–filler adhesion in the composites, silane coupling agents are widely used for surface treatment of fillers, especially inorganic oxide fillers, such as silica, alumina

Great concern regarding environmental issue and the non-renewable of petroleum has attracted many researchers to research about biocomposite plastic. Thus the purpose of this research is to produce biodegradable biocomposite and good mechanical properties film using renewable source by produce by biopolymer composites from starch and alumina.

2. Literature review

a) Composites matrix of starch

Starch has been studied for several decades due to its availability, biodegradability and lower cost. Starch is composed of linear amylose and branched amylopectin, and is considered the main form of stored carbohydrates in plants such as rice, potatoes or corn (Cynthia et al 2014). Starch can show a thermoplastic behavior if water, glycerol or sorbitol is added as plasticizer. In spite of its many advantages, starch based materials have severe disadvantages due to poor process ability, weak mechanical properties, poor long-term stability and high water sensitivity (Tang et al 2012).

These polysaccharides have been studied combined with synthetic or natural polymers: starch-polyethylene, starch-henequen and starch-coconut (Aguilar et al 2007), starch-poly(lactic acid), polyhydroxyester ether, or poly(hydroxybutyrate-co-valerate) (Willett and Schrogen, 2002), poly(vinyl alcohol)-chitosan (Hang et al 2010), chitosan-poly(lactic acid) (Bonilla et al 2013) as well as starch-chitosan (Mathew et al 2006).

(Xiaofei et al 2005) stated that the matrix of natural fiber of thermoplastic starch (TPS) was prepared with urea and formamide as the mixed plasticizer. This TPS could effectively restrain starch retrogradation. SEM micrographs showed good dispersion and adhesion between starch and fiber. Studies in the dependence of mechanical properties of reinforced TPS on the contents of fiber and water demonstrated that with increasing fiber content from 0 to 20%, the initial tensile strength was trebled up to 15.16 MPa, while the elongation was reduced from 105 to 19%. The reinforcement effect was gradually weakened with the increasing of water contents, but at the high water contents (O30%) both the fiber and water contents had no effect on the tensile strength.

(Yaret et al 2010) In this work the synthesis and mechanical characterization of a polymer matrix composite is reported. An epoxy resin is used as matrix with addition of starch and coconut fibers as reinforcement. Vickers hardness and impact tests are used for mechanical characterization. Starch is used to promote degradability of the polymer matrix with clear benefits for the environment. Natural fibers have been used for reinforcing the composite materials. Natural fibers have several advantages such as price, low density and relatively high mechanical properties, they are also biodegradable and non-abrasive. In one such study, starch was reinforced with bacterial cellulose, and the tensile properties, resistance to biodegradation, and moisture absorption were studied.

b) Composites filler of metal (alumina)

(Kobayashi et al 2000) Three composites consisting of alumina powder dispersed in a bisphenol-a-glycidyl methacrylate (Bis-GMA) matrix were prepared and evaluated to assess the effect of alumina powder content on the mechanical properties and osteoconductivity of the composite. The alumina powder composites (APC) consisted of alumina powder (AL-P) as the inorganic filler dispersed in a Bis-GMA matrix that was solidified by a radical polymerization process. Prior to polymerization the AL-P was mixed with the monomers in proportions of 50%, 70%, and 80% by weight (APC50, APC70, and APC80). A fused silica-glass-filled composite containing 70% glass by weight (SGC70) was used as a control. The compressive and bending strengths, the elastic modulus in bending, and the bending strain of the composites increased as the AL-P content increased

(Noraiham et al 2008) studied that mechanical properties of Epoxidised natural rubber (ENR) filled various loading of alumina nanoparticles from figure 1.

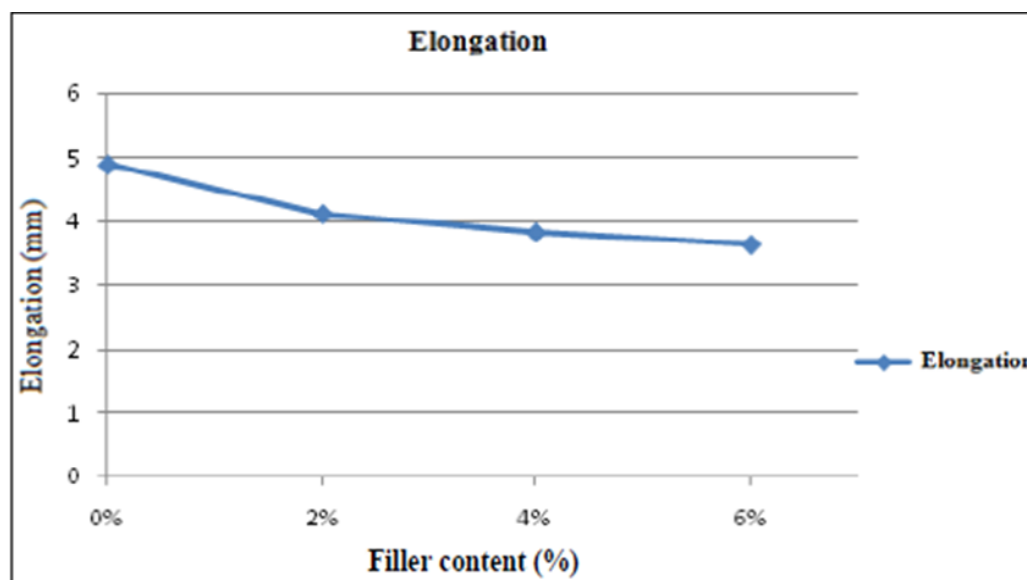
Figure 1: mechanical properties of Epoxidised natural rubber (ENR) filled various loading of alumina nanoparticles (Noraiham et al 2008)

Properties	Alumina Nanoparticles (phr)						
	0	10	20	30	40	50	60
Tensile strength (MPa)	20.93	17.35	17.16	16.45	16.36	15.71	14.80
Tensile modulus (MPa)							
100% Elongation	0.63	0.74	0.75	0.97	0.99	1.15	1.34
300% Elongation	2.68	2.94	3.70	4.16	4.26	4.96	5.44
Elongation at break, EB (%)	625	557	540	540	525	521	495
Hardness (Shore A)	11.7	12.5	13.63	14.43	15.73	17.13	18.93
Impact Strength (J/m)	239.75	215.55	175.24	172.39	168.74	168.48	153.95

The increase in hardness is related with high tensile modulus and the increasing amount of hard alumina nanoparticles in the ENR matrices. On the other hand, as can be suggested from the impact test, the elastic behavior of the matrix proportionately varies with the addition of the alumina nanoparticles. As the loading of alumina fillers increases, the ability of the composites to absorb impact energy decreases since there are less ratio of the rubber matrix to fillers. This result in decreasing impact strength which explained the capability of the composites to withstand energy before fracture.

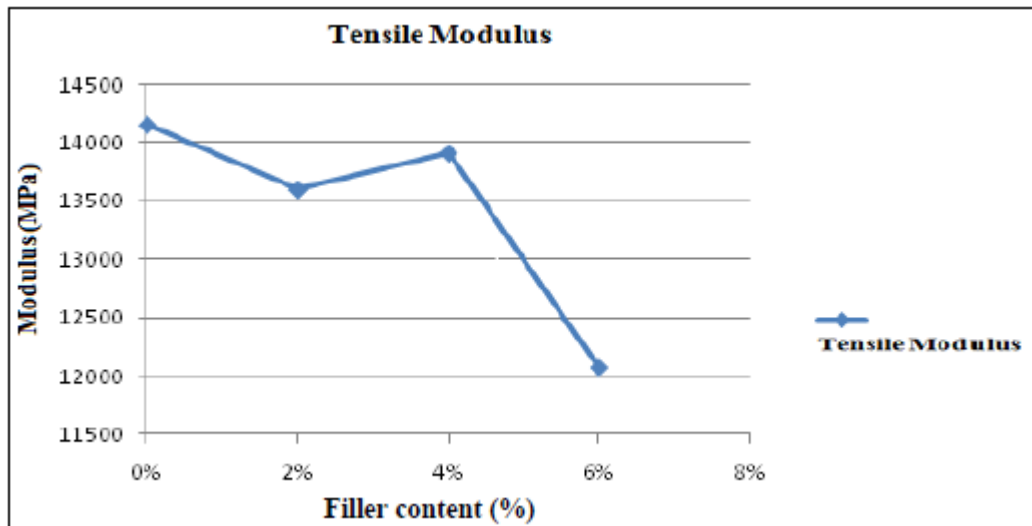
The effect of addition of alumina-graphite fillers on tensile properties of glass fabric reinforced epoxy resin composites has been evaluated experimentally. The results of elongation as a function of filler content of neat epoxy and their composites are shown in Figure 2. For all the composites tested, it is observed that the elongation decreases linearly with increase in filler content (Puneeth et al 2015).

Figure 2: Elongation v/s filler content%. (Puneeth et al 2015).



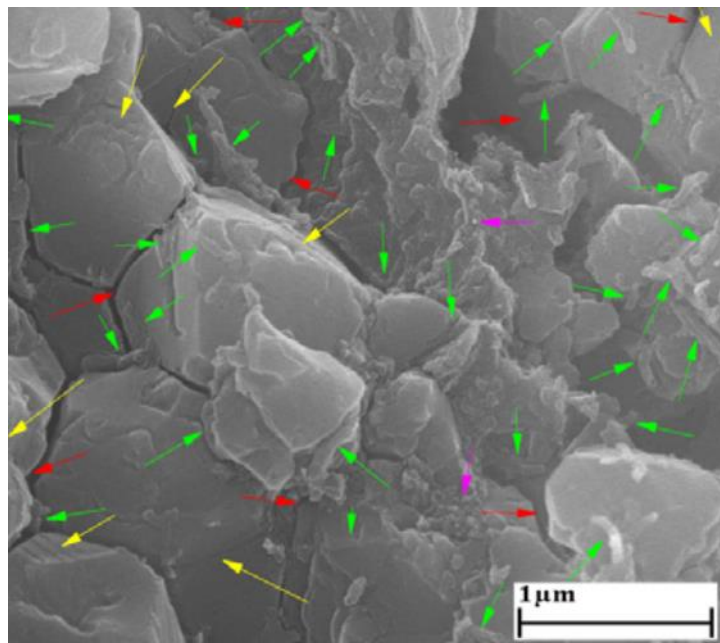
The results of Tensile Modulus as a function of Filler Content of neat epoxy and their composites are shown in Figure 3. For all the composites tested, it is observed that the tensile modulus decreases with increase in filler content.

Figure 3: Tensile modulus v/s filler content%. (Puneeth et al 2015).



(Yi et al 2015) from the fracture behavior of a carbon nanotube (CNT) and carbon alumina composites at cryogenic temperature (77K) is reported. CNTs are well dispersed and embedded in the alumina matrix (shown by green arrows).

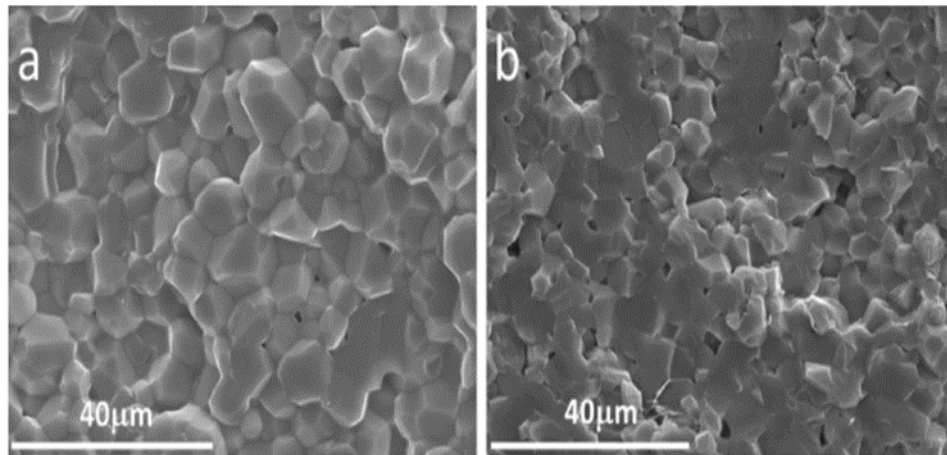
Figure 4: Morphology of the fracture mode of the CNTs/Al₂O₃ composite at 77 K. (Yi et al 2015)



These CNTs Effectively bond with Al₂O₃ grains, resulting in crack paths deflection. Moreover, the mixed intergranular (shown by red arrows) and transgranular (shown by yellow arrows) fracture modes of CNTs/Al₂O₃ composite are clearly evident. Simultaneously, transgranular fracture also occurs for most alumina grains, which indicates that the interface between CNTs and Al₂O₃ grains can effectively transfer alien loads (figure 4). In addition, it is also found from fractographic analysis that a part of CNTs wrap with fine Al₂O₃ grains at the grain boundaries and form a few shear deformation zone (shown by purple arrows), which effectively hinder crack propagation and facilitate crack paths deflection.

(Bahareh et al 2014) Alumina nanocomposites reinforced with hybrid GNTs (graphene nanoplatelets, GNPs, and carbon nanotubes, CNTs) were fabricated by hot-pressing. In Fig. 5a and 5b, the fracture morphology was found to change from intergranular mode to trans-granular mode. The finer grains can lead to improved hardness and strength of the composites, due to the grain boundary pinning effect which impeded the dislocation movements This means that the GNT addition has also made the grain boundaries stronger, which would lead to other toughening mechanisms for the improved properties.

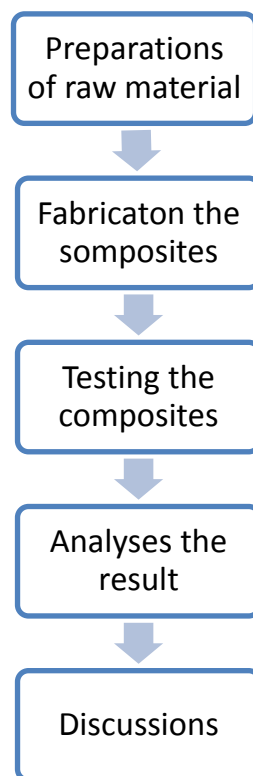
Figure 5: SEM images of fractured surfaces: (a) monolithic Al_2O_3 exhibiting an inter-granular fracture mode, (b) S0.5–1 showing a trans-granular fracture mode (Bahareh et al 2014)



3. Material and experimental method

The methodology that involves for each activity are preparations the sample composites, testing and analyses the sample. Each of the methodology activity will explain for more understanding. Refer figure 3.1 for the flow chart of research methodology

Figure 3.0: Research flow chart of research methodology



3.1 Raw material use

The preparation of the sample composite as figure 6. The list of material (alumina nanoparticle, water, starch and glycerol) that use to form the new nanoparticle alumina starch composite. This preparation have done by manually. (Refer appendixes part for more details)

Figure 6: List of material uses (alumina nanoparticle, water, starch and glycerol)



3.2 Fabrication of Alumina Nanoparticle – starch composite

The composites preparations mixing of Alumina Nanoparticle (Al₂O₃), starch, water and glycerol (CH₂OH.CHOH.CH₂OH). In this mixing, the amount that fixed are water and glycerol and amount that manipulated are starch and alumina nanoparticle (Al₂O₃). This for the purpose for studying what is the effect of alumina nanoparticle to the composites. The measurement unit in centimeter which are width = 1.5 cm, long = 10 cm and thickness= 0.02 cm (figure 7).

Table 1: Material weight distribution

Sample	Water (g)	Glycerol (g)	Sample No	Starch (g)	Alumina Nanoparticle (g)
A	100	2.5	A ₀	5	0
			A ₁	4.7	0.3
			A ₂	4.4	0.6
			A ₃	4.1	0.9
			A ₄	3.8	1.2
B	100	2.5	B ₀	10	0
			B ₁	9.7	0.3
			B ₂	9.4	0.6
			B ₃	9.1	0.9
			B ₄	8.8	1.2
C	100	2.5	C ₀	15	0
			C ₁	14.7	0.3
			C ₂	14.4	0.6
			C ₃	14.1	0.9
			C ₄	13.8	1.2

Figure 7: After cutting the composite



3.3 Alumina – starch composites testing

The determination tensile of composite by following ASTM International D 3039/D 3039M. The summary of this test method is thin flat strip of material having a constant rectangular cross section is mounted in the grips of a mechanical testing machine and loaded in tension while recording load. The ultimate strength of the material can be determined from the maximum load carried before failure tensile properties of polymer matrix composite materials. Figure 8 shows while conducting the EZ test Shimadzu testing.

Figure 8: EZ test Shimadzu testing



3.4 Tensile test failure codes/typical modes

Mode and location failure of the specimens by using three-part failure mode that is shown in figure and table

Figure 9: Tensile test failure

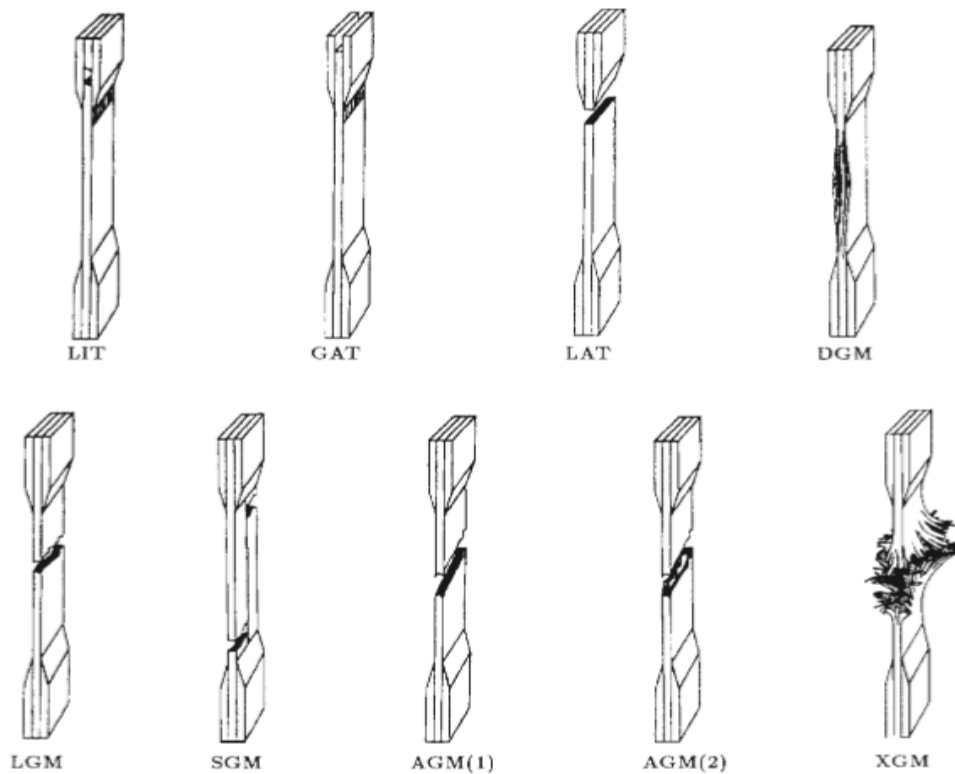


Table 2: Failure and code type

Failure type	Code	Failure area	Code	Failure location	Code
Angled	A	Inside grip/tab		Bottom	
Edge/Delamination	D	At grip/tab		Top	
Grip/tab	G	<1W from grip/tab		Left	
Lateral	L	Gage		Right	
Multi-mode	M(xyz)	Multiple area		Middle	
Long splitting	S	Various		Various	
Explosive	X	Unknown		Unknown	
Other	O				

3.5 Scanning Electron Microscopy (SEM) JSM-5610 LV

The Scanning electron microscopy JSM-5610 LV, featuring a wide application microscope, incorporates the functions of a High Vacuum SEM and Low Vacuum SEM in a single instrument. The LV SEM allows nonconductive specimens to be observed in

their original. Since it has no need to lower the accelerating voltage, it allows elemental analysis with an energy dispersive X-ray spectrometer. It also gives opportunity for the observation of specimens that contain water or are stained with oil.

Figure 10: Scanning Electron Microscopy (SEM) JSM-5610 LV



4. Result and discussion

4.1 Tensile test failure/typical modes

Refer from tensile test failure and code on experimental methods after finished done the testing, there are several type of failure showed such as Lateral gage middle (LGM), Angled gage middle (AGM) and Lateral at grip/tab top (LAT). Standard test method D 3039/D 3039M (Tensile properties of polymer matrix composite material) stated the factors should include such as tab alignment, tab material, tab angle, tab adhesive, grip type, grip pressure and grip alignment. (Jane et al 2005) was verified no occurrence of failure by shear and/or debonding in the interface between laminate/tab. Therefore, all the occurred failure modes are considered valid and used to calculate the tensile strength and modulus of the tested specimens.





Table 11: Lateral gage middle (LGM)



Table 12: Angled gage middle (AGM)

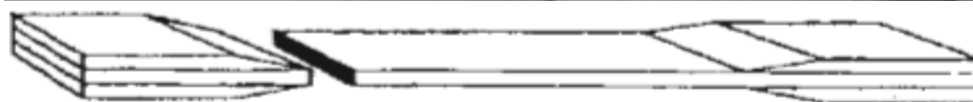
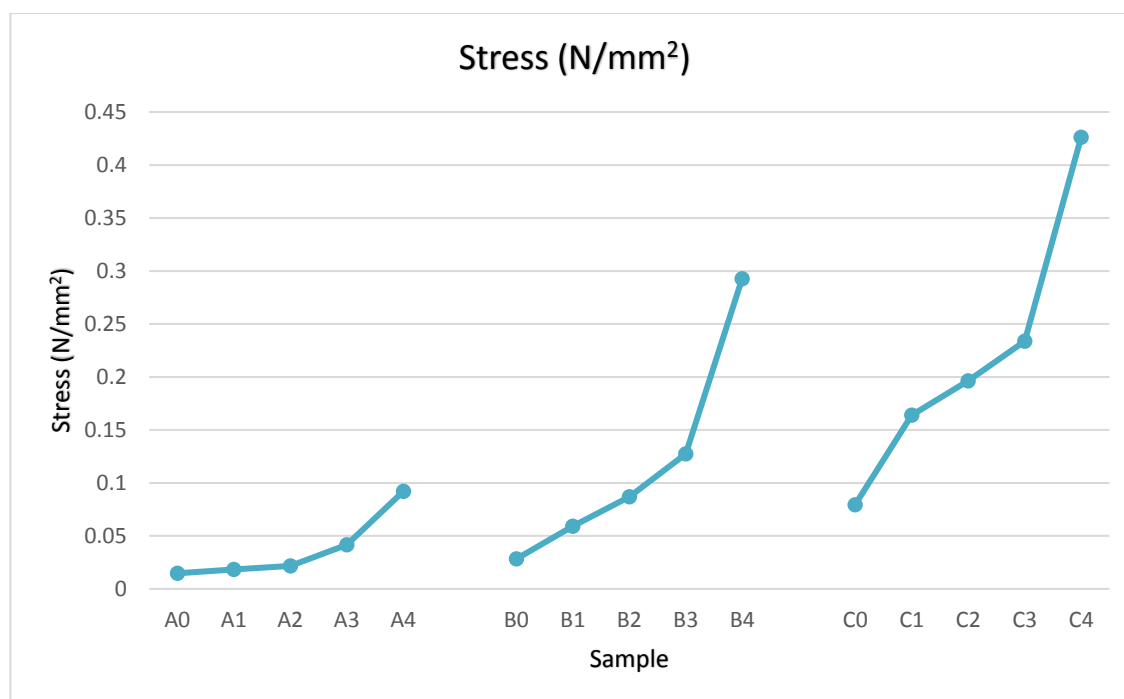


Table 13: Lateral at grip/tab top

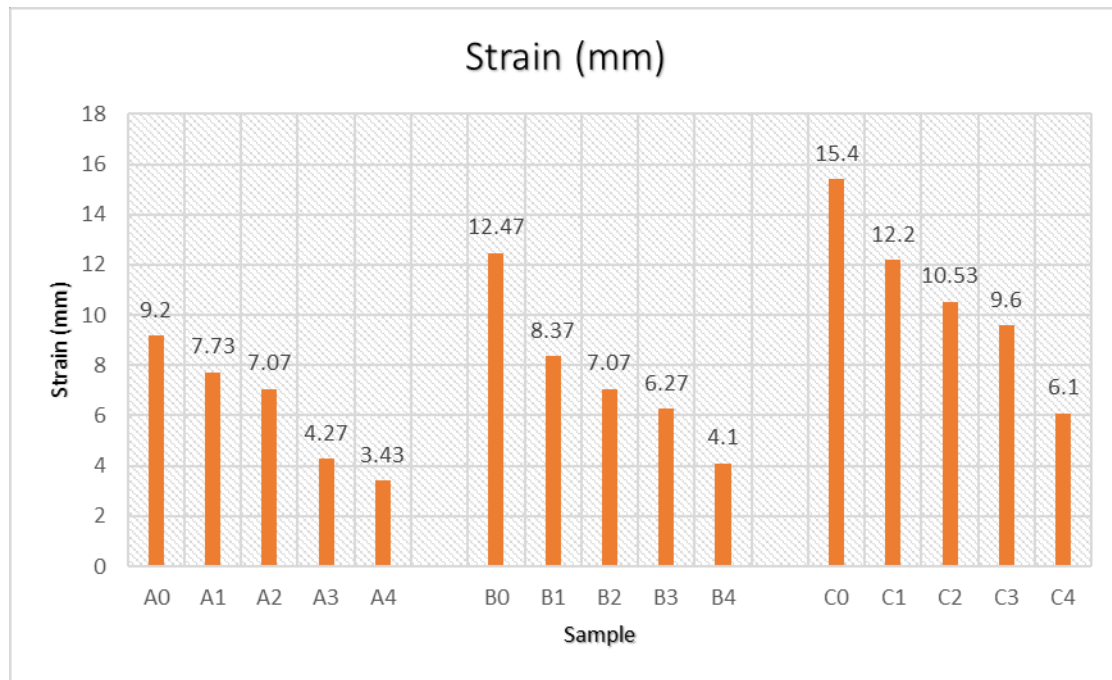
4.2 Strain (mm) and Stress (N/mm²) and Scanning Electron Microscope (SEM) analysis

Figure 14: Stress (N/mm²)



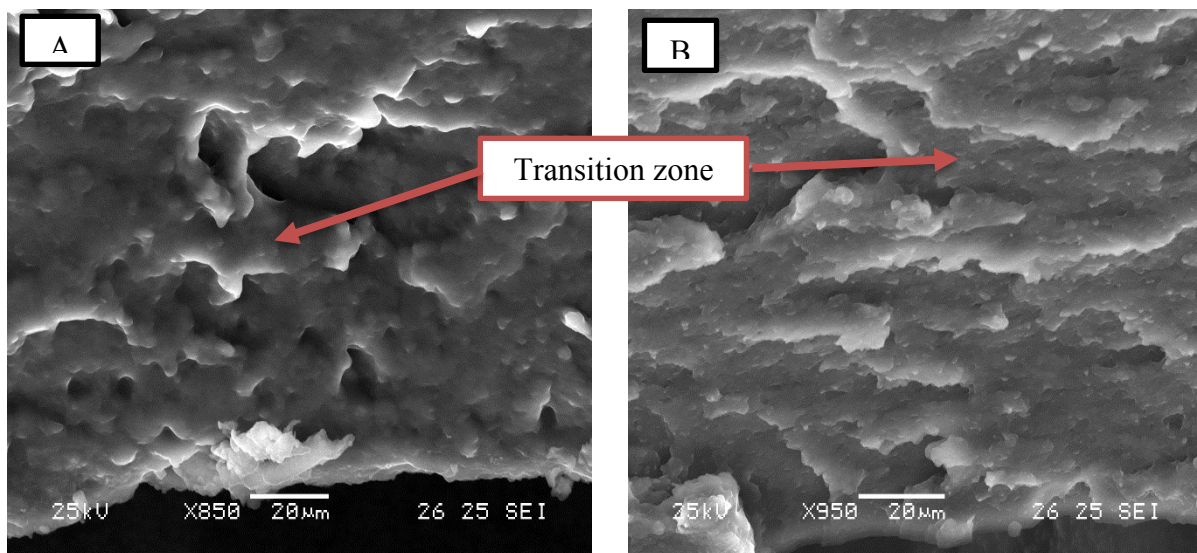
Based on stress graph, three sample labelling as A (A0, A1, A2, A3, A4), B (B0, B1, B2, B3, B4) and C (C0, C1, C2, C3, C4) with different weight distribution of raw material as table 1. By increasing alumina nanoparticle (Al_2O_3) as nanofillers (reinforcement) to the tapioca starch, the tensile properties increase directly with it before the composite start to crack. The exact reason for this variation can be attributed to the fact that alumina particles play like stress concentrators, and this role is promoted as particles lead to be agglomerated. Thus, during tensile loading, the magnitude of stress increases drastically near the agglomerated nanoparticles (Arezou et al 2015). The values of agglomeration of filler particles play an important role in creation of stress concentration and cause cracks propagate faster so fracture occurs immediately (Hauptert and Wetzel, 2005). Alumina is one of the most widely used engineering ceramic materials due to its high elastic modulus, high wear resistance and chemical corrosion resistance, high-temperature stability and the retention of strength at high temperatures (Raju et al 2014). However, the drawback of common alumina is its poor mechanical properties, such as flexural strength (about 380 MPa) and fracture toughness (about $3.5 \text{ Mpa}^{1/2}$) (Munro, 1997). Recently, a bending strength up to 654 MPa and a fracture toughness up to $5.7 \text{ Mpa}^{1/2}$ have been obtained for alumina ceramics by using low-temperature-sinter able high purity alumina powder (Rao et al 2000) stated that alumina will be more widely used for engineering applications if its mechanical properties can be further improved. So, from the graph by combination with tapioca starch as composite increasing the mechanical properties. Finally, as expected the lower the starch content the higher the strength for all composites. This supported by (Emrullahoglu et al 2013) the higher the starch content the lower strength of composites.

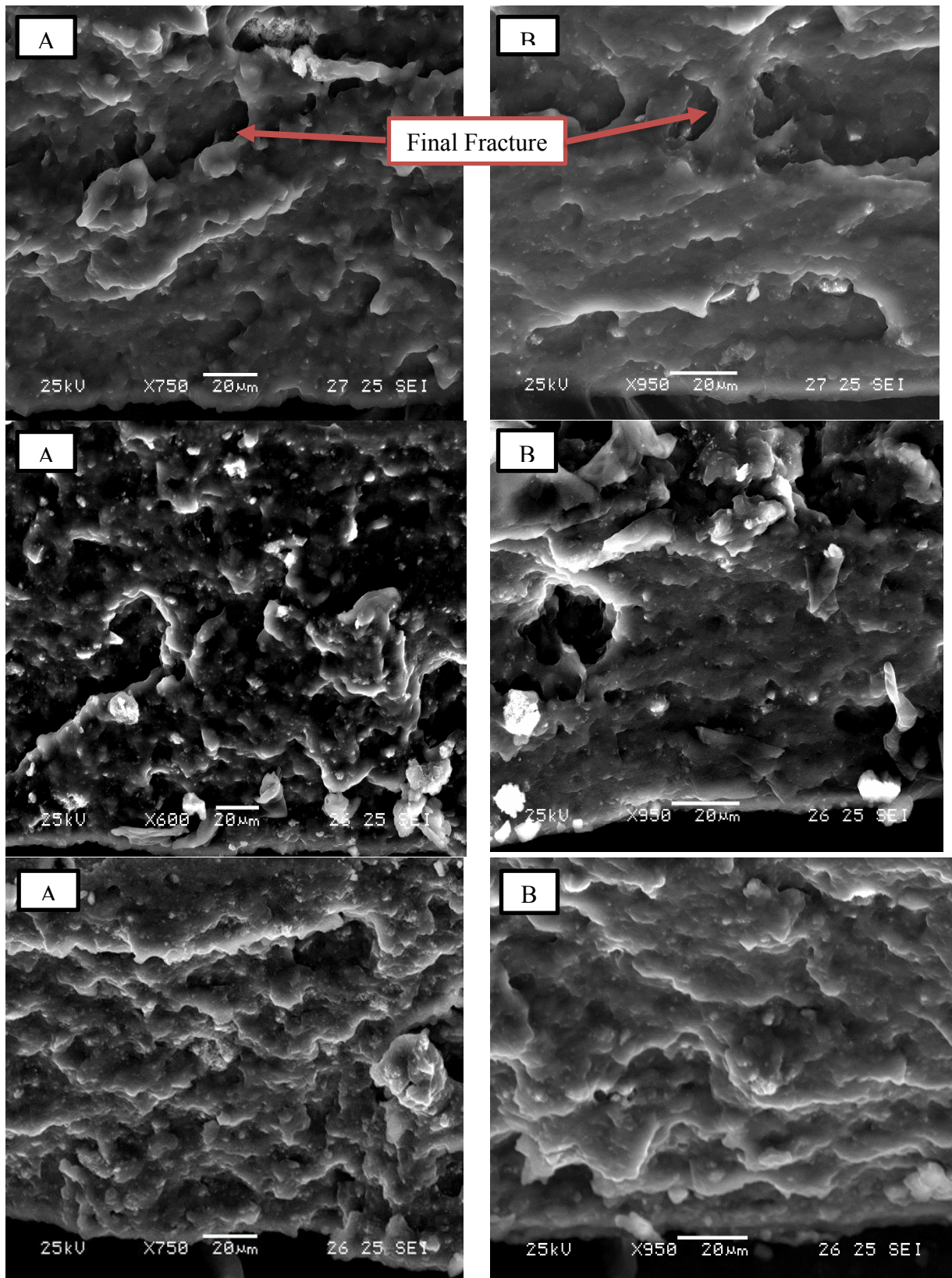
Figure 15: Average elongation (mm)

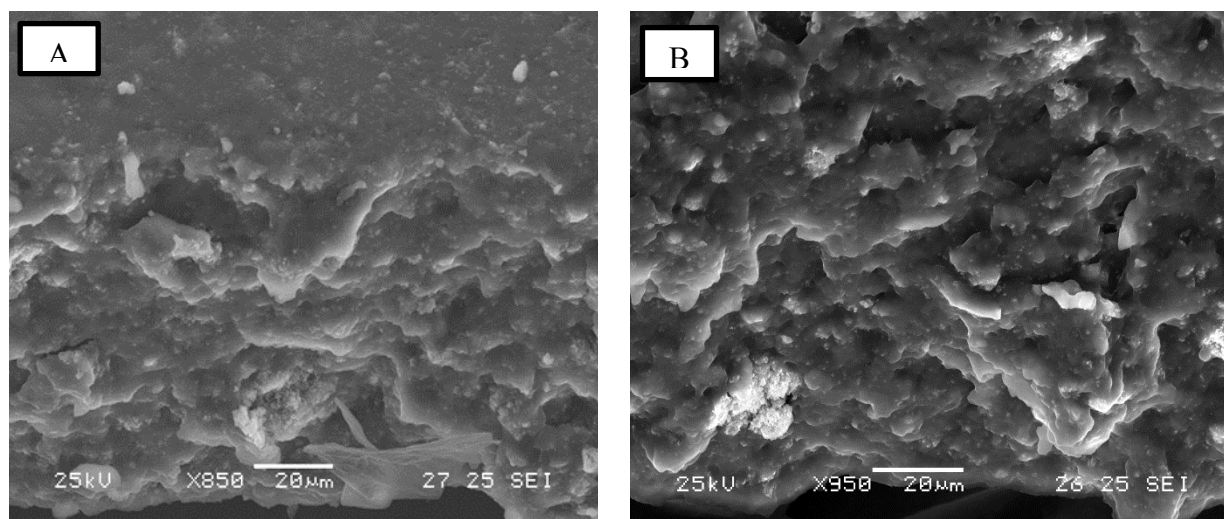


From the strain analysis above, the elongation three sample labelling as A (A0, A1, A2, A3, A4), B (B0, B1, B2, B3, B4) and C (C0, C1, C2, C3, C4) is decreasing with increasing the weight of the alumina nanoparticle (Al_2O_3). This elongation strong related to the tensile strength, when tensile strength is increase the elongation of the composite decrease. Thus, the elastic properties of starch are decrease when it reinforces to alumina nanoparticle (Al_2O_3).

Figure 16: Scanning Electron Microscopy images







Based on the figure 16 for sample as A (A0, A1, A2, A3, A4), B (B0, B1, B2, B3, B4) shows the composite crack creation. (Areazou et al 2015) stated the rate of creation cracks of sample due to increase of applied stress cause to change the morphology of fracture surface. By increasing the weight distribution of alumina nanoparticle, the particle bonding between tapioca starch and alumina nanoparticle become stronger. It shows by figure 16, whereas the images that transform from the particle bonding much closer by increasing the weight alumina nanoparticle (Al_2O_3). Thus, need more applied stress to break the bonding between tapioca starch and alumina nanoparticle. In fact also, by addition of nano alumina (Al_2O_3) causes to increase pseudo voids due to creation of more debonding. (Almeida et al 2003) explained that the fracture surface of thermoset polymers can be characterized by the presence of three different regions including a flat featureless mirror zone surrounding the crack initiation point, a transition zone, in which the surface roughness steadily increases, and a final propagation zone with conical marks. The surface final propagation (fracture occurred) is much higher than that of the transition zone.

5. Conclusion

Tapioca starch reinforced by alumina nanoparticle (Al_2O_3) were prepared successfully by casting on leveled trays. The properties of the starch alumina composite were improved by increasing weight percentage to the composite. The results showed good mechanical properties and also supported by the SEM analysis. Starch materials are poor process ability and properties (e.g. weak mechanical properties, poor long-term stability, and high water sensitivity). The addition alumina nanoparticle (Al_2O_3) to form new composite had been seen the increasing their properties.

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