Incorporation of coconut shell based nanoparticles in kenaf/coconut fibres reinforced vinyl ester composites

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Abstract

In the present study, a successful attempt has been made on enhancing the properties of hybrid kenaf/coconut fibers reinforced vinyl ester composites by incorporating nanofillers obtained from coconut shell. Coconut shells were grinded followed by 30 hours of high energy ball milling for the production of nanoparticles. Particle size analyzer demonstrated that the size of 90% of obtained nanoparticles ranged between 15 to 140 nm. Furthermore, it was observed that the incorporation of coconut shell nanofillers into hybrid composite increased water absorption capacity. Moreover, tensile, flexural, and impact strength increased with the filler loading up to 3 wt.% and thereafter decrease was observed at higher filler concentration. However, elongation at break decreased and thermal stability increased in nanoparticles concentration dependent manner. Morphological analysis of composite with 3% of filler loading showed minimum voids and fiber pull outs and this indicated that the stress was successfully absorbed by the fiber.

Keywords: kenaf fiber, coconut fiber, coconut shell nanoparticles, vinyl ester, hybrid composite

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1. Introduction

Natural plant fibers have good quality in terms of strength, low density, and wide availability besides that they are also environmental friendly owing to their biodegradability [1]. Thus they are widely researched for fabrication of composite materials. Commonly used natural fibers are hemp, jute, flax, kenaf, oil palm empty fruit bunch, bamboo, and coconut fiber. Among them, kenaf is well known for its ability to produce strong fiber and have potential to replace glass fiber [2]. Kenaf (*Hibiscus cannabinus L.*) is one of the fastest growing annual plants that can reach up to a height of 3-4 meters in 120-150 days. It is an excellent source of cellulose fibers and suitable for reinforcement in composites [3]. Composite materials generally consist of two or more phases namely matrix and reinforcement. It utilized the advantages of all added components to overcome the weakness of individual components. Chin and Yousif [4] fabricated composite by using epoxy resin and kenaf fibers for bearing applications. In another set of study suitability of different natural fibers like kenaf, coir, sisal, and hemp as reinforcement in polypropylene matrix were evaluated and the results obtained indicated that kenaf fiber/polypropylene composite had the highest strength and tensile modulus among all studied samples [2].

Beside kenaf fiber, coconut based fiber are also widely researched for development of composite material. Coconut (*Cocos nucifera*) is one of Arecaceae species which is same as palm family. Every part of coconut palm is of potential usefulness including coconut shell and coconut coir fibre which is lignocellulosic in nature. The fraction weight percentage of coconut coir fibre is 30-40% and coconut shell is 15-20% of coconut fruit [5, 6]. Coconut shell is of low economic value and usually considered as agricultural waste. Recently, many researchers suggested that coconut shell and coconut fibres have excellent strength properties and thus are

suitable to be used as reinforcement in composite materials [7-9]. Aireddy and Mishra [10] investigated coconut dust reinforced epoxy matrix composite and it was observed that abrasive wear resistance increased with coconut dust concentration. Furthermore coconut shell nanofillers are also widely researched for nanocomposite preparation owing to their high aspect ratio. Pradhan *et al*. [11] reported that the incorporation of upto 20 wt.% of coconut shell powder in ultrahigh molecular weight polyethylene resulted in improved mechanical properties. Madakson *et al*. [12] stated that addition of coconut shell ash as filler in composite enhanced its mechanical and physical characteristics.

Mechanical properties of vinyl ester resin are similar to epoxy resin and it can easily be handled at room temperature. However, chemical resistance, hydrolytic stability, and cure rate of vinyl ester are better than polyester [13]. It also has the ability to cure at room temperature, has a low viscosity which introduces high wettability of matrix towards the reinforcing additives [14], and low price [15]. Various types of natural fibers including bamboo, kenaf, flax, sisal, banana, jute, coir, oil palm EFB, pineapple leaf, and bagasse as reinforcement in vinyl ester polymer composites for improvement of mechanical properties has been studied in past [16-18]. Furthermore, it was also reported that the addition of nanoclay in vinyl ester composites led to improved thermal stability [19].

Besides numerous advantages of natural fiber, their shortcomings like low melting point, lack of interfacial adhesion, low modulus elasticity, and rapid degradation process or aging towards humidity make them less attractive to be used as reinforcement in the polymer composite. In order to overcome this problem nanoparticle has been identified as suitable material that can enhance the properties of natural fibre reinforced polymer composite. Nanoparticles of oil palm ash (OPA) and oil palm shell (OPS) is widely used as filler in

biocomposites, however, a study on using coconut shell nanostructure as fillers is very limited. Therefore, in this study, coconut shell nanoparticles was produced and used as filler to enhance properties of kenaf/coconut coir fiber reinforced vinyl ester composite.

2. Experimental details

2.1. Preparation of coconut shell nanoparticles

Coconut shell was obtained from Kampung Tanjung Sari, Teluk Intan plantation site located in Malaysia. Hammered coconut shell was grinded by a wiley mill and sieved by 60 mesh size to separate the micro-sized particle from the mill such as stone, sand, and other impurities. Obtained particle was dried in an oven at 110 °C for 24 hours followed by crushing and sieving to a particle size fraction of 2 to 2.8 mm. Later, acquired samples were grinded again for four cycles using the 0.25 mm MF sieves. Afterward, coconut shell powder was grinded by using high energy ball milling for 30 hours at 170 revolutions per minute with w/w ratio of balls to powder was 10:1. Toluene was added into the ball mill having an anionic surface active agent to prevent the agglomeration of coconut shell powder [20]. After milling samples were oven dried at 110 °C for 24 hours. Obtained nanoparticles were then transferred into a thimble and placed into a soxhlet extraction unit connected to a reaction flask containing 350 mL of n-hexane solution. The extraction was carried out for 8 hours followed by drying in an oven at 105 °C for 24 hours and cooling in a desiccator. Finally, nanoparticles were grinded in high energy ball milling for another 2 hours to prevent its agglomeration after the extraction process.

2.2. Particle Size Analysis

Particle size distribution of nanostructured coconut shell was assessed on a Zeta Sizer Malvern Ver 6.11 (MAL 1029406, Germany) by using dynamic light scattering measurements by means of a 532nm laser. The measurement of the average particle size was performed in triplicate.

2.3. Preparation of hybrid composite

Different concentration of nanofiller 0, 1, 3, and 5 wt.% (of resin) was slowly added into vinyl ester resin (Zam Scientific & Supplies Sdn. Bhd, Malaysia). The resultant mixture was stirred at 3000 rpm for 5 minutes followed by addition of methyl ethyl ketone peroxide (MEKP, 1.5 wt.% of resin) (Zam Scientific & Supplies Sdn. Bhd, Malaysia) as a catalyst. Obtained mixture was stirred at 200 rpm for 5 minutes and then cobalt naphthenate (0.2 wt.% of resin) as an accelerator was added.

Trilayer hybrid nanocomposite was prepared by soaking three layers of fiber mat kenaf-coconut-kenaf (K-C-K) in above prepared resin solution until all fibers got wet. Kenaf mat of dimension 200mm × 200mm was obtained from National Kenaf and Tobacco Board Malaysia (Kota Bharu, Kelantan) and coconut coir fiber mat in non-woven form with the same dimension was provided by Ecofiber Technology Sdn. Bhd (Malaysia). Stainless steel mold with the dimensions of 200mm × 200mm × 5mm was sprayed with silicon and then the wet trilayer fiber mat was put into it and cold pressed at 200psi later mold was left to cure at room temperature for 24 hours. Hybrid composite without nanofillers was coded with K-C-K/0, 1% was coded with K-C-K/1, 3% with K-C-K/3, and 5% with K-C-K/5.

2.4. Density, Void, and Water Absorption Measurement

The density of fabricated composites was measured according to ASTM D1895 (1996) while the volume of the samples was measured by using a digital vernier caliper (MItutoyo). ASTM D

2734 (1996) method was used for determination of the void content of composites. Water absorption test was carried out according to ASTM D570. In detail, square size samples with dimensions of 20mm × 20mm × 5mm were cut from composites. Then, the samples were weighed to get the initial weight and subsequently immersed in the water at room temperature. After 24 hours, samples were taken out and were gently blotted with filter paper to remove excess water from surface and finally samples were weighed again. This process was continued for several days until a constant weight of samples were achieved. Five samples were tested from each composite.

$$\text{Water Absorption } (\%) = \frac{Wn - Wd}{Wd} \times 100$$

where, W_n = the weight of composite samples after immersion, W_d = the weight of the composite samples before immersion.

2.5. Tensile Test

The tensile test was performed according to ASTM D3039 (2000) using Instron Model 5582 Universal Testing Machine. Each sample was cut into rectangular shape with the dimension of 120 mm × 15 mm × 5 mm. The gauge length was set at 60mm with the crosshead speed of 5 mm/min. Tensile properties including tensile strength, tensile modulus, and elongation at break were acquired from data produced. Five replicates for each type of composite were tested.

2.6. Flexural Test

The flexural test was conducted using Instron Model 5582 Universal Testing Machine according to ASTM D790 (2003) specification. Each sample was cut into rectangular shape with the dimension of 160 mm × 20 mm × 5 mm. Five replicates for each type of composite were tested.

Testing was carried out through three point bend test at room temperature with a crosshead speed of 2 mm/min.

2.7. Izod Notched Impact Test

The Izod notched impact test was conducted according to ASTM D256 (2006) using Geotech Testing Machine, Model GT-7045 MD. Each sample was cut using a circular saw with the dimension of 70 mm × 15 mm × 5 mm. Five replicates of each composite were tested.

2.8. Morphological Analysis

Fractured surface during flexural and impact test was analyzed using scanning electron microscope (Leo Supra, 50 VP, Carl Zeiss, SMT, Germany). The surface of specimens was coated with a thin gold palladium layer using Sputter Coater Polaron SC515 to prevent the surface charge of specimens during electron beam exposure. Using double sided tape specimens were bonded on scanning electron microscopy (SEM) holder. SEM micrograph was determined under conventional secondary electron imaging conditions at 5kV of acceleration voltage.

2.9. Thermogravimetric Analysis

The thermogravimetric analyzer was used to investigate the thermal stability of composites by employing Perkin Elmer Pyris 1 TGA with Pyris software version 9.1.0.0203. The analysis was conducted in temperature ranges from 30 to 800 °C with a heating rate of 20 °C per min under nitrogen atmosphere.

3. Results and discussion

3.1. Particle size

It was observed that the size of 90% of produced nanoparticles ranged between 15 to 140 nm with irregular shapes having no agglomeration (figure 1). Based on the previous study by Dungani *et al*. [21] variation in particle size was a result of ball milling. Coconut shell nanoparticles have good distribution and excellent dispersion according to Petrovicova [22].

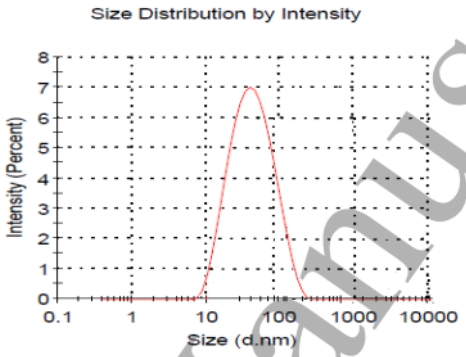

Figure 1. Particle size distributions of coconut shell based nanoparticles.

3.2. Density, Void, and Water Absorption Analysis

Density and void content of developed composites were presented in Table 1. Theoretical and measured density of all studied samples increased in nanoparticles concentration dependent manner. Composite without any nanofillers has a theoretical density of 1.1375 g/cm^3 and for measured density, it was 1.0745 g/cm^3. Both increased to a value of 1.1595 and 1.0915 g/cm^3 for theoretical and measured density, respectively. Moreover, it was also observed that nanoparticles incorporation up to 3% resulted in a decrease in void content from 5.538% of composite without nanofillers to 4.88%. This might be due to the fact that nanoparticles effectively occupied the voids within samples. Further increase in nanofiller concentration to 5% led to an increase in void content to a value of 5.864% due to agglomeration of nanoparticles at higher concentration.

Table 1. Physical properties of nanofillers reinforced kenaf-coconut-kenaf fibre based vinyl ester composite.

Filler Loading (Wt. %)	Theoretical Density (g/cm³)	Measured Density (g/cm³)	Void Content (%)
K-C-K/0	1.1375	1.0745	5.538
K-C-K/1	1.1415	1.0835	5.08
K-C-K/3	1.1435	1.0875	4.88
K-C-K/5	1.1595	1.0915	5.864

It is very important to examine the water absorption behavior of composite because mechanical properties and dimensional stability depends on its moisture absorption capacity. There are several factors that can affect water uptake capacity of composites such as process of formation of composite, and types of filler, fibre, and matrix [23]. Figure 2 shows the water absorption of hybrid K-C-K fibre incorporated with nano filler. It was observed that the water absorption ability increased with the amount of nano filler loading. This might be due to the hygroscopic nature of cellulosic material of filler [24]. All samples showed increasing trend of water uptake in beginning of the immersion but slowly went down and reached the saturation level after 264 hours of immersion. Hybrid composite with 5% filler loading (K-C-K/5) had highest percentage of absorbed water which was 7.24% and composite without filler loading (K-C-K/0) had 6.15% of water absorbed.

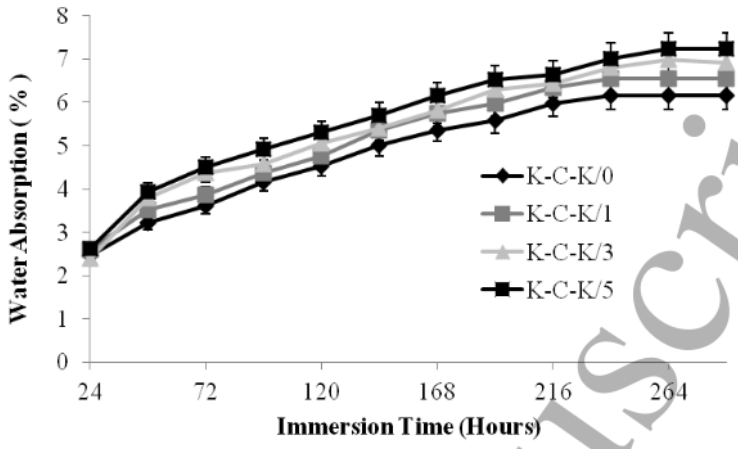

Figure 2. Water absorption capacity hybrid kenaf-coconut-kenaf fibre reinforced vinyl ester nanocomposites.

3.3. Tensile Strength

Tensile strength is referred as the capability of a material to resist forces that tend to pull it apart. Figure 3 presents the tensile strength of nanostructured coconut shell filled hybrid K-C-K fiber reinforced vinyl ester composite. It was clearly seen that the hybrid composite without filler had lowest tensile strength among all samples with a value of 47.66 MPa. The addition of nanofillers in composites resulted in an increased in tensile strength up to 3% of filler loading with concentration dependent decrease thereafter. The tensile strength of hybrid composite at 3% of filler loading was 58.69 MPa and at 5% it was reduced to 51.27 MPa. In the previous study, highest tensile strength was achieved at 3% of nanofillers loading in epoxy composites was reported [25]. The observed increase in tensile strength was due to better interaction between filler, matrix, and fiber because of coconut shell nanoparticles. This resulted in the high interfacial area between fillers and matrix which led to wettability enhancement in composites [26]. Increased surface area made vinyl ester resin capable of transmitting and distributing stress

within samples when force applied. However, tensile strength decreased at 5% of filler loading due to agglomeration of nanoparticles in composites at higher concentration.

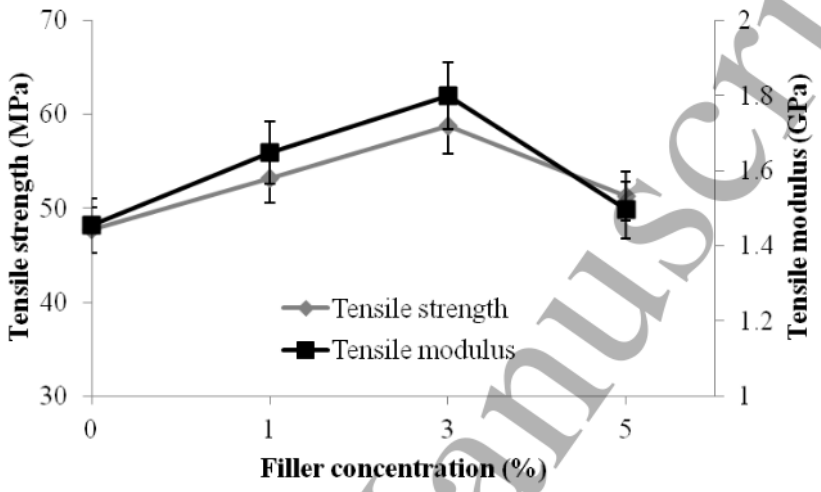

Figure 3. Effect of nanoparticles loading on tensile strength and tensile modulus of kenaf-coconut-kenaf fibre reinforced vinyl ester composite.

3.4. Tensile Modulus

Tensile modulus is the ability of a material to extend and undergo stretch before breaking. Figure 3 shows the effect on tensile modulus when nanofillers were incorporated in hybrid K-C-K fiber composite. Tensile modulus increased from 1.45 GPa to 1.8 GPa when nanoparticles concentration increased from 0% to 3%, respectively, and thereafter it decreased to a value of 1.49 GPa at 5% of nanofillers. In the previous study, Sarki et al. [6] also reported that the addition of nanofillers increased tensile strength and tensile modulus of composites. Nanoparticles play an important role to stiffen the vinyl ester resin since rigid organic particles generally have a much higher stiffness than polymer matrix [27]. Thus, the stress would be transferred from resin to the fiber and showed a higher value of tensile modulus of the developed

composite as compared to sample without nanoparticles. Furthermore, at 5% agglomeration of the nanofillers occurred which resulted in a reduction of tensile modulus.

3.5. Elongation at Break

Figure 4 shows the elongation at break of all samples. It was observed that elongation at break decreased with increase in nanostructured filler loading. %Elongation decreased to a value of 4.39% from 5.18% when nanofillers concentration increased from 0% to 5%. Similar trend was observed by Mohaiyiddin et al. [26]. The observed reduction in elasticity was due to the higher rigidity of coconut shell based nanoparticles than that of vinyl ester resin. This increased the brittleness of hybrid composites and reduced the ability of the matrix to elongate as its mobility was restricted by organic nanoparticles [28].

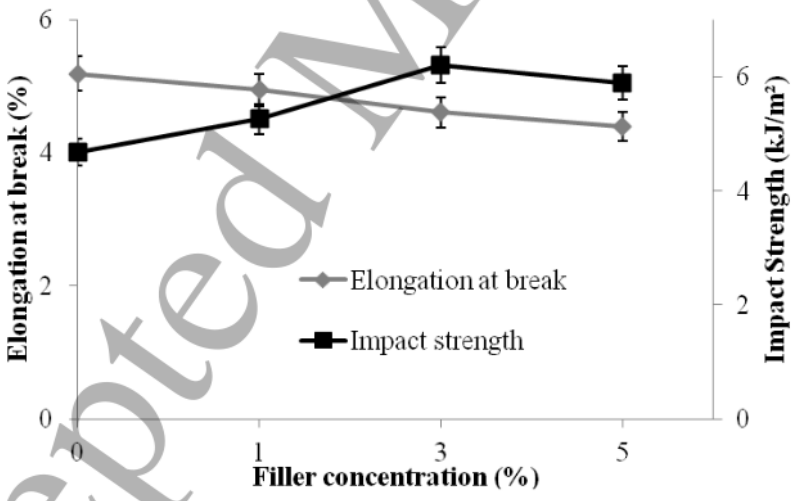

Figure 4. Effect of nanoparticles loading on elongation at break and impact strength of kenaf-coconut-kenaf fibre reinforced vinyl ester nanocomposite.

3.6. Flexural Strength

Flexural strength is a mechanical parameter used to identify the ability of a material to withstand bending forces that applied on its longitudinal axis. Figure 5 shows the flexural strength of fibre reinforced vinyl ester composite. Incorporation of nanoparticles resulted in enhancement of flexural strength of composites. Hybrid composite without filler showed the lowest flexural strength of 68.85 MPa and for hybrid composite with 3% of nanofillers it reached a maximum value of 87.23 MPa. High polarity of resin helps in the formation of strong hydrogen bond with hydroxyl group thus increase in flexural strength of the composites was observed. However, higher filler concentration increases the viscosity of matrix which subsequently triggers internal porosities and reduce its strength to a value of 75.18 MPa at 5% of nanoparticles in composites [29]. Results obtained in present study were similar to previous findings [29, 30].

3.7. Flexural Modulus

Flexural modulus is stiffness during initial bending. Figure 5 shows flexural modulus of nanoparticles reinforced vinyl ester composites. It was observed that the addition of nanofillers increased flexural modulus of hybrid composite. Highest flexural modulus of 4.82 GPa was demonstrated by composite incorporated with 3% filler and for samples, without any filler, it was 3.85 GPa. Homogeneous dispersion of coconut shell nanoparticles leads to an improvement in interfacial adhesion between nanofillers and vinyl ester matrix which triggered restriction in the flexibility of polymeric chains [31]. Further increase in coconut shell filler loading to 5% led toward a reduction in flexural modulus to 4.2 GPa.

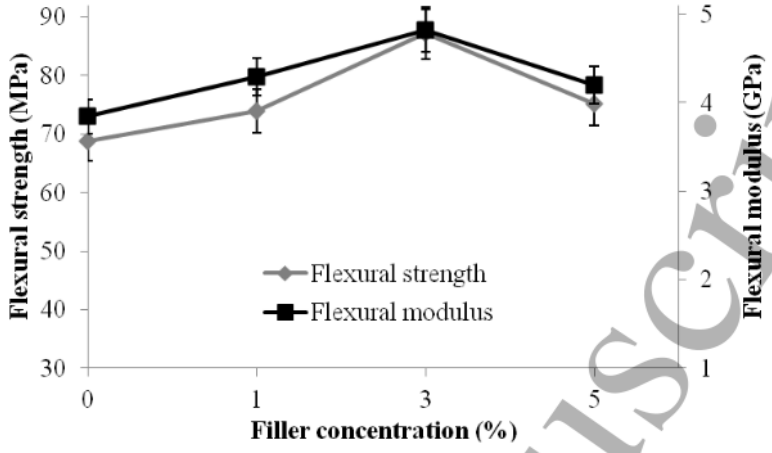

Figure 5. Effect of nanoparticles loading on flexural strength and flexural modulus of kenaf-coconut-kenaf fibre reinforced vinyl ester nanocomposite.

3.8. Izod Notch Impact Test

The impact test is defined as the capability of a material to withstand fracture under stress applied at high speed and it is directly related to overall toughness. Figure 4 shows the effect of different filler loading (0, 1, 3 and 5 %) on impact strength of composites. Composite without nanoparticles demonstrated impact strength of 4.67 KJ/m^2. It can be seen that the impact strength of hybrid composite increased with filler concentration and the highest impact strength was noted at 3% filler loading with a value of 6.21 KJ/m^2. Thereafter, it decreased to 5.89 KJ/m^2 for 5% of filler loaded composite. Coconut shell nanofillers has porous surface hence it effectively binds with matrix resin during composite fabrication and thus enhancement in impact strength was observed. Furthermore, the energy from the impact was absorbed easily and crack propagation was prevented this improved the impact strength of tested samples. However, with further increase in filler concentration decrease in impact strength was demonstrated by composite sample due to agglomeration of nanoparticles.

3.9. Scanning Electron Microscopy

Figure 6 shows SEM image of fractured surface of composite samples after impact testing. Nanoparticles cannot be seen in any micrograph images this was due to the nanosized filler having the measurement from 15 to 140 nm and it was well dispersed within the matrix. It was apparent from obtained images that the occurrence of fibre detachment and voids formation in composite without filler was caused by weak bonding between fiber and matrix. However, addition of 1% of nanofillers in hybrid composite slightly improved the mechanical strength due to well formed matrix around fibers but fiber pullouts and small gaps between fibers and matrix were still present. Composite with 3% of filler showed smaller and lesser voids as compared to other samples. Moreover, well formed matrix around fibers further indicated good interfacial bonding between fiber and matrix which resulted in the better transfer of applied stress from the matrix to fiber and this led to an increase in mechanical strength [32]. Void content increased at filler loading of 5% and this indicated that higher filler concentration caused weak wetting of fiber. Large voids around fibers are prominent due to agglomeration of nanoparticles and this is further able to serve as a point of initiation failure [33]. Thus SEM image analysis explained observed improvement in mechanical properties of developed composites due to nanoparticles incorporation up to 3%.

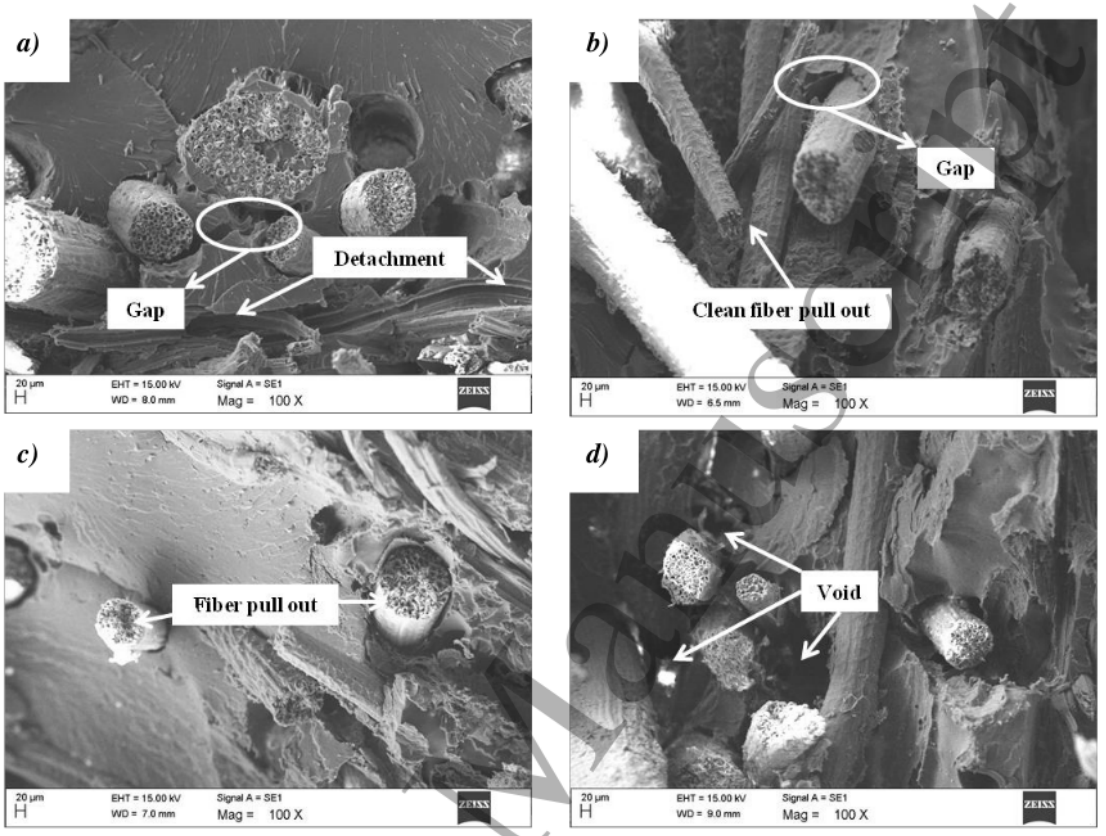

Figure 6. SEM micrographs of vinyl ester composite reinforced with kenaf-coconut-kenaf fibre having nanofiller concentration: (a) 0%, (b) 1%, (c) 3%, (d) 5%.

3.10. Thermal Properties

Figure 7 shows the thermal decomposition curves of the composites and it was observed that all samples show two stages of degradation process. First stage of degradation was the weight loss of samples below 100°C due to moisture removal [34]. Second stage of degradation was observed at temperature ranges from 250 to 550 °C due to removal of volatile compounds from samples at higher temperatures.

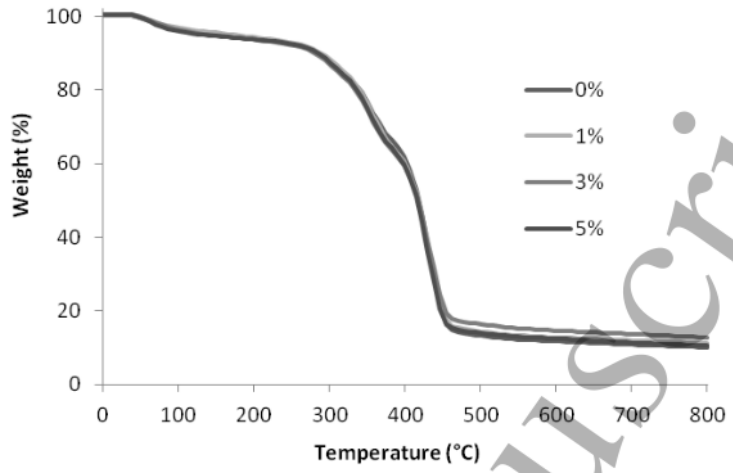

Figure 7. Effect of nanoparticles loading on thermal stability of kenaf-coconut-kenaf fibre reinforced vinyl ester nanocomposites.

In present study, $T_{10\%}$ (T_{onsett}) and $T_{50\%}$ were determined which signifies 10 and 50 wt.% weight loss, respectively. T_{50}% is considered as structural destabilization point of the tested sample. T_{10}% and T_{50}% of all composites were tabulated in Table 2. Both degradation temperatures of composite with nanofillers were higher than the composite without filler. Results showed that the thermal stability of samples increased with the nanofillers loading. Inter-particle interaction between coconut shell nanoparticles and matrix protects and consume much heat from the composites hence improvement in thermal stability was observed [35]. Furthermore, char residue of composites increased in nanoparticles concentration dependent manner due to inorganic and nonvolatile compounds present in nanofillers (Table 2).

Table 2. Thermal properties of different nano filler reinforced in vinyl ester composite.

Composite	Degradation Temperature (°C)		Char Residue (%)
	$T_{10\%}$	$T_{50\%}$	
K-C-K /0	306	409	32.44
K-C-K /1	308	410	32.61
K-C-K /3	311	412	35.62
K-C-K /5	314	415	37.01

4. Conclusion

In the present study, the effect of various percentages of nanofillers loading in hybrid kenaf/coconut coir fiber reinforced vinyl ester composites on physical, mechanical, thermal, and morphological properties were studied. Incorporation of 3% of nanofillers resulted in improved mechanical properties of composites including tensile strength, tensile modulus, flexural strength, flexural modulus, and impact strength. This was due to the incorporation of high density coconut shell nanoparticles in low density vinyl ester matrix. Nanoparticles tend to fill the voids and led to the reduction of void content in the hybrid composites. Furthermore, the morphological study of SEM showed the reduction in fiber pullouts from vinyl ester matrix as the nanoparticles loading increased to 3% thus explained the observed enhancement in various characteristics of composites. However, mechanical properties were decreased upon incorporation of 5% of nanoparticles due to agglomeration of fillers which resulted in large voids formation as confirmed by SEM image analysis. The decline in elongation at break in the nanofillers concentration dependent manner was observed for all composites. It was also observed that the filler incorporation resulted in enhancement in thermal stability of composites. Overall, it can be said that coconut shell nanoparticles were effectively used as filler in hybrid kenaf/coconut fiber reinforced vinyl ester composites and the optimum filler loading was demonstrated at 3%.

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