Effect of kenaf fibre Mat layers and Zinc Oxide Nanoparticle Concentration on the Mechanical and Thermal Properties of ZnONPs/kenaf/polyester Composites

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Abstract. Zinc oxide nanoparticle kenaf polyester composite (ZnONPs/kenaf/polyester) containing 1, 2, 3 and 4 layers of kenaf fibre treated with 40nm 2% (w/w) and 5 % (w/w) of ZnONPs were prepared and characterised to evaluate the mechanical and thermal properties. The composites were prepared based on 4 kenaf layers and 2%, 5% (w/w) ZnONPs concentrations, all the samples were compared with control systems containing zero kenaf fibre and untreated with ZnONPs to save as references. The composites were fabricated using cold mixed blend via compression sandwich mould technique. The composites with 1 layer 5% (w/w) ZnONPs treated had the lower tensile strength and lower modulus suggesting it higher flexibility compared with those systems containing high number of layers, similar trend were seen in 2% (w/w) treated ZnONPs concentrations. The mechanical properties of composites containing 5% (w/w) ZnONPs demonstrated that the addition of fibre layers to the ZnONPs/kenaf/polyester was affected more than the 2% (w/w). While thermogravimetric analysis of 5% (w/w) and 2% kenaf composites incorporated with 1 and 4 layers were equally compared. The (5%) ZnONPs/kenaf/polyester treated revealed no significant changes in the decomposition temperature and showed no thermal transitions, this suggesting that it is non-biodegradable. Both results of the mechanical and thermal properties test suggest that the 1% (w/w) provide the based mechanical and thermal stable composite with novel biodegradability.

1. Introduction
The fibre based biodegradable polymer composite materials with good mechanical and thermal properties has attracted the attention of research communities recently (The trend interest is expected to grow in daily basis as the materials have high potential for various applications [1-10]. Due to this requirements, there is need to improve it degradability [11-18]. This degradability is improved by treated and several treatment approaches have been proposed for example [6-8] have proposed hydrogen peroxide on Kenaf Fiber/Poly(Lactic Acid) Composites, several have been reported such increase in crystallinity index and surface roughness of the kenaf fiber which improved mechanical
properties of the prepared composite, however, this in essence of removing some vital parts in the composite such as lignin and hemicellulose[19-30]. Moreover, proposed reinforcement of kenaf unsaturated polyester composite with glass fibre, the study enhanced the adhesion between the surface of fibre and the matrix which that allowed improving the mechanical properties of the KG-UPE hybrid composites prepared, however, this might come with getting the composite more toward nonbiodegradability [31-40]. Several researchers proposed alkaline treatment on kenaf/epoxy composite and found alkali treatment increased the mechanical properties of the composites by allowing improvement of fiber–matrix compatibility, similarly, the chemical treatment bring about lignin and hemicellulose removal or degradation. The compatibility between fibre–matrix interface adhesion is the most significant parameter that determine the properties of composites [41-45]. One of the important issues related to natural fiber is the hydrophilic property of cellulose which has an impact on the weak interface bonding with hydrophobic polymer as a matrix[46]. The methods of chemical surface modifications of natural fiber have been proposed by many studies, this include chemical treatment such alkaline most of the this treatment removes lignin and hemicellulose [47-49]. however, this have overall effect on the overall mechanical properties of the composite, the search for the alternative materials have occupied the mind researcher in the field. Recently zinc oxide have been proposed to been used in killing bacteria. The treatment mechanism are as follow, immersing the the zinc ion (Zn²⁺) into aqua ions, it formed water ligands with hexa and tetra-aqua ions, the reaction of the ion with hydroxide through donation of a proton on the surface of the fibre [1,2, 46-51].

2. Methods
Kenaf fiber was obtained from RahamatullahSdn. Bhd, Malaysia. The kenaf were in the form of mat and used without any further treatment. A standard unsaturated polyesters resin was supplied by Castmesch Technologies Sdn. Bhd. MEKP(solution in dimethyl phthalate) used was from KaumjungAkzoNobel peroxide Ltd by trade name Butanox M60. Nano-zinc oxide (ZnO,<100 nm particle size)was purchased from Sigma Aldrich Chemistry, USA. nano-zinc oxide (nano-ZnO) and The unsaturated polyester (UP), methyl ethyl ketone peroxide (MEKP) UP has hazy and pinkish color in appearance with gel time 18 to 23 minutes at 25 °C with 2% MEKP. Density of UP was 1.4 with specific gravity 1.12 g/cm³ and volumetric shrinkage at 8%. MEKP has colorless in appearance. Density of MEKP was 1.15 with melting point -8 °C and boiling point at 109°C. The fabrication and testing consist of 7 majors steps: Initial, the Kenaf fiber mat was cut to 20 cm × 20 cm dimensions. Different concentrations of nano-ZnO were applied, i.e. 2 and 5 wt%. High-speed mixing employed a Ragogna mixer custom built for FPInnovations by Custom Machinery Ltd. (speed up to 5,000 rpm). To avoid destabilization of the emulsion, a moderate mixing speed (up to 2,500 rpm) at room temperature. A good dispersion was obtained with powder. The followed by submerging the prepared kenaf into prepared nanoparticle in water, the process according to the procedure in (Mohammed et al, 20017). To avoid sedimentation of nanoparticles , Kenaf fiber was immediately submerged in the suspensions containing nano-ZnO for 60 min at 60 °C and at a vacuum of 600 mm Hg to increase penetration of the solution. The treated kenaf fibers were then washed with distilled water to remove the excess chemicals.

3. Results and Discussion
The composite tensile properties were determined based on tensile stress, modulus of the elasticity and tensile modulus. All the parameters determine the mechanical properties of the composite and determine greatest amount of stress a sample can withstand before failure occurred. Four different systems containing different layers arrangements of the kenaf mats composites are tested against two concentrations for tensile stress and modulus of the composites. The results of the mechanical test indicated slightly linear for all curves and the point of deviation from linearity indicates failure initiation in kenaf layer which is bit contrary to what claimed by others. Working with natural material provide several advantages, it offers high specific strength and modulus, low cost, low density, renewable nature, biodegradability, absence of associated health hazards.
To study the mechanical properties of the composite and its tensile strength changes, the tensile stretched morphology of all the 4 layers were studied at 2% and 5% treatment concentrations were analyzed using atomic force microscopy (AFM) technique. Figure 1a-c shows the surface morphology of 1 layer, 2 layers, 3 layers and 4 layers respectively. As can be seen in the figure2 c and d there are small particles which extended upward, these as results of some parties still did not penetrated to kenaf. Moreover, the surface morphology of 3 and 4 layers kenaf fiber also became rougher and textured. A rough fiber surface can create good interlocking with the matrix surface and give good unsaturated-kenaf fiber adhesion. This can be proved from the tensile modulus composite which shows larger strength as the number of the layers increased. In other hand, as the percent of zinc oxide increase poor interfacial adhesion can be seen due to the encouragement of wide bonding gap between fibre and polyester. It is important to note that improvement in mechanical properties of the polymeric composites can result from good compatibility between fiber and matrix as explained in the above equation.

Figure 1. Show kenaf fibre with (a) 1 layer (b) 2 layers (c) 3 layers (d) 4 layers
Figure 2. (a) FTIR spectrum of zero layers kenaf composite, 1 layer kenaf composite, 2 layers kenaf composite, 3 layers kenaf composite and 4 layers kenaf composite (b) Tensile stress–extension curve for the prepared composites

The figure 2a shows the FTIR spectra of pure saturated polyester (0 layers), 1 layer, 2 layers, 3 layers and 4 layers. The FTIR spectra of the fibres indicate that intense peak at 950 CM\(^{-1}\) for 1300 CM\(^{-1}\) for zero layers and 4 layers composite compressed suggests chain-chain hydrogen-bonded reducing of chain stretching, rotation and flexibility through vibration from the cellulose and lignin structure of the mats. The peaks in the range 1500–2000 CM\(^{-1}\) are related to the chain in hemicelluloses and lignin stretching in unsaturated polyester. The peaks beyond 2000 CM\(^{-1}\), especially at 2500 CM\(^{-1}\) show a tiny peak characteristic stretching vibration from in cellulose and hemicellulose. The presence of peaks at the 3000 CM\(^{-1}\) and beyond may be because of the stretching of acetyl groups of hemicellulose. The figure 2b was plotted based on tensile stress testing of ZnONPs/kenaf/polyester Composites. It shows typical tensile extension curves for different kenaf layer mats. 0 kenaf layer (only unsaturated polyester, 1 layers kenaf, 2 layers kenaf, 3 layers kenaf and 4 layers kenaf, unsaturated matrix material was also tested for comparison purposes. This figure clearly shows that the ZnONPs/kenaf/polyester Composites becoming more rigid, and stronger as the no of the mats increase. More so, the figure show the stress vs strain curves of the composite based on the different layers of the kenaf mats layers. The 4 layers kenaf shows highest stress. However, it shows decreased tensile strength, this due to the strongly bound with the polymer matrix resulted from natural fibre affinity to unsaturated polyester. This eventually allow uniform distribution of fibres intimate contact
between the particles and the matrix (Zhanhu et al, 2008). Moreover, as the number of the mat layers decrease the tensile strain increase, this is due to the stress concentration is lower and the stresses can be more easily transferred from the matrix to the fibres. The stress between the fibres and the matrix propagate concentric forces across the composite which eventually elongate the molecular chain of the matrix. However, this elongation is decrease with the number of the kenaf mat layers from 4.3% to 3.1%, it caused by the restriction of the molecular chain elongation and reduce share thinning.

![Graphs showing modulus of elasticity and tensile stress of kenaf composites](image)

**Figure 3.** (a) Modulus of elasticity of kenaf composites containing: 1 layer, 2 layers, 3 layers and 4 layers (b) Tensile modulus of the composites containing 1 layer (Untreated, 2%treated, 5%treated), 2 layers (Untreated, 2%treated, 5%treated), 3 layers (Untreated, 2%treated, 5%treated); 4 layers (Untreated, 2%treated, 5%treated) (c) Tensile stress of the composites containing 1 layer (Untreated, 2%treated, 5%treated), 2 layers (Untreated, 2%treated, 5%treated), 3 layers (Untreated, 2%treated, 5%treated); 4 layers (Untreated, 2%treated, 5%treated)

In order to identify the effluence of Kenaf mat fibers layers and the treatment by the zinc oxide nanoparticle the tensile modulus of composite were determined using tensile test and presented here. The mechanical properties of the untreated, treated with 2% and 5% and the with different fibre mats are shown. The composites treated with 25 with show increased tensile modulus compared to the counter part with by the 5% treated composite. It also evident that in figure 6, the kenaf layers from 1 to 4 layers, the Modulus of elasticity of kenaf composites formulations containing 3 and 4 layers are higher by up to 25% and 35% respectively compared with the neat 1 layer compared. In also evident that, the natural fiber in general and kenaf fibre specially act not only to fill the polymer intermolecular gaps but also provide reinforcement for the composite by providing hammock to molecular chain movement as a results damping is achieved. According to Intan et al, 2014 The reinforcement may occur through a mechanism in which the applied stress is transferred from one fibre to the next thereby enabling an even distribution of the stress throughout the material. From the figure 2 it is evident, the surface is rough, which allowed the increase adhesive characteristics and thus facilitate the stress transfer during an applied load due to the good mechanical interlocking between the kenaf fibres and the matrix. The rough surface of kenaf fibre results from the removal of which
created as a result of the present Nano particles. The figure 7 presents the stress against the treated and untreated composite with different layers of kenaf layers arrangement. Tensile modulus increases linearly with the increase of the kenaf layers. With maximum tensile modulus of 2450PMa by the 2% treated 4 layers kenaf with lowest tensile modulus of 1400PMa by the 5% 1 layer kenaf composite. Generally tensile modulus linear behaviour. The results further revealed that the increase in kenaf fiber layers resulted in a reduction of the impact strength of the composites. Moreover, a continuous decrease in impact strength also can be observed as the fiber composition was increased from 2 to 5%. However, when compared to 2% and 5% treated composites, 2% composites showed improvement in impact strength. In the treated nano particles absorbing mechanism depends on the interfacial interaction between fiber and the particles as illustrated in previous sections. Therefore, it is believed that the increase in zinc nano particle improve in fiber-matrix adhesion. However, does not exceed to 2% concentration. The tensile modulus of the composites containing 1 layer Untreated show 1500PMa with 2% treated produced slightly higher with 1560PMa however, with 5% treated showed reduction might be due to the fact that increase beyond 2%, the there is no enough hydroxyl group to enhance solubility of the zinc nano particles thus agglomeration is formed, in 2 layers system, the untreated composite show 1750PMa, 2% treated produced 1780PMa, 5% treated produced 1770PMa, in 3 layers Untreated composite produced 2200PMa while 2% treated 2170PMa and 5% treated, the 4 layers Untreated produced 2350PMa while 2% treated produce 2340PMa and for 5% treated 2320PMa. Thus, the kenaf layer decrease tensile modulus, therefore It can be said that kenaf fibre has a great impact and influence on tensile modulus properties and the treated with zinc nanoparticle treatment can further enhance the properties of composite.

4. Conclusion
The ZnONPs/kenaf/polyester Composites based on unsaturated polyester and 4 difference layers kenaf treated with ZnONPs were prepared and characterized. The optimum conditions for the zinc oxide nanoparticles and kenaf mats layers treated unsaturated polyester nanocomposites have been studied. 1% Zinc oxide nanoparticles treated and 4 layers kenaf composite were found to favor the nanocomposite thermal and mechanical properties. The treatment with lower concentration increases the adhesion matrix to the kenaf natural fibre. The observed increased thermal stability and improved mechanical properties in the composites treated with ZnONPs nanoparticles are closely attributed to the good nanoparticle dispersion in the polymer matrix and to the introduced chemical bonding. The nanocomposites become mechanically stable with higher no of kenaf mats layers as compared to the lower kenaf layers, the effect of kenaf layers are independent of the ZnONPs concentration.

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