



Effect of Nano-Filler Content and Fiber Treatment on the Characteristics of Gamma-irradiated Rice Husk-Epoxy Particleboard Composites

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Received 13th Sept.
2018
Accepted 30th Jan. 2019

In this study, both untreated and treated rice husk fibers with a silane coupling agent are incorporated with epoxy resin to prepare particleboard composites. The effect of different ratios of nanoclay namely, 5%, 10%, 15%, and 20% (by the weight of the polymer) on the physicochemical properties of the prepared particleboard composites is investigated. The results show that the flexural, and impact strengths as a function of mechanical properties getting better with increasing nanoclay content up to 10%. While the addition of nanoclay to the samples decreases the water absorption percentage. This decrease is almost constant with nanoclay (%). In addition, the effect of different doses of gamma-irradiation on the properties of both untreated and treated rice husk-epoxy particleboard composites that containing 10% of nanoclay is also discussed. The results show that the irradiated specimens at a dose of 10 kGy achieved a good enhancement in mechanical properties whereas the water absorption percentage as a physical property decreases at a dose of 10 kGy and starts to increase with increasing gamma irradiation doses. TGA, and SEM are carried out to confirm the data obtained.

Keywords: Nanoclay/ Epoxy resin/ Gamma-irradiation/ Particleboard composite/ TGA

Introduction

As a result of increasing the awareness of the importance of interconnectivity of global environmental factors such as principles of sustainability, green chemistry, eco-efficiency, and industrial ecology, many efforts are being made in this area. For this reason, natural fibers have recently gained a great interest to be used as reinforcements and fillers in polymer composites due to their several advantages such as low cost, low density, low energy consumption, high specific properties, besides their biodegradable ability [1, 2]. Every year, a huge amount of rice husk fibers, as an agricultural waste, is removed and separated from rice grains during rice milling

[3]. So, many researchers have tried to develop its applications and to minimize the environmental problems caused by it. Recently, cellulosic fibers such as rice husk, bamboo, wood, paper, and sisal are considered alternative materials that can be used for making particleboard since the production of industrial wood from natural sources has been decreased continuously as a result of the lack of the use of forest areas in industrial production and its transformation into recreational areas. With the increasing population, it is also necessary to try to find alternative resources to replace wood raw materials [4-6]. Precisely, rice husk is used as a reinforcement for the production of particleboard composites due to their good specifications such as

cheapness, lightness, high porosity, high surface area. This is in addition to the fact that their ash contains a high percentage of silica and they need low energy during manufacturing [7]. In general, the use of renewable biomass as a raw material in particleboard composite production may result in several environmental and economic benefits as it reduces the rates of pollution resulting from the burning of the agricultural remnants and raises the level of public health as well as low weight, low production cost, and good mechanical properties. Also, it is considered an insulating material which is useful as a reinforcing agent in building industry [8-10].

The utilization of rice husk as a natural fiber in particleboard composite materials requires a strong adhesion between the fiber and the matrix since the performance and the mechanical properties of natural fiber-polymer particle board composites depend mainly on the interphase properties which are responsible for transferring the stress between the fibers across the matrix [11]. The major problem in natural fiber-polymer composites is the poor interactions between the hydrophilic natural fibers and the hydrophobic polymer matrix which leads to a considerable decrease in the properties of the composites [12, 13]. This is due to the presence of cellulose as one of the components of natural fiber with a polar surface associated to hydroxyl groups (OH) which is responsible for the hydrophilic nature of the fibers and as a result cellulose forms strong hydrogen bonds between fibers. The hydrophilic hydroxyl groups are removed from the fiber by the effect of different chemicals. For this reason, the surface modification of the fibers by chemical treatments is necessary to reduce the hydrophilic nature of the fibers and to improve the adhesion between fibers and polymer matrix [14-16]. These chemical treatments depend on the use of reagent functional groups that are capable of reacting with fiber structures and changing their composition and surface morphology [17].

Improving final performance properties of particleboard composites using nanotechnology application in manufacturing of particleboard composite is of a great importance. Only limited work in the literature discussed developing particleboard composites reinforced with nanoparticles such as nano SiO₂, nano Al₂O₃, nano

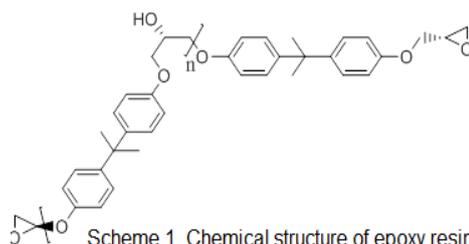
ZnO, nanoclay and other nanomaterials. Recently, mixing of natural fiber with mineral additives has been used to improve the physical and mechanical properties of particleboard composites [18]. Compared to micro-particles, nanoparticles allow some unparalleled properties of polymer composites as the large surface area of nanofillers promotes better interfacial interactions with the polymer, leading to better property enhancements [19-21]. Generally, introducing nanofillers into natural fiber-polymer composites may be profitable for enhancing their properties. However, a homogeneous dispersion of nanoparticles in a polymer is very important to achieve those improvements [22].

Utilization of ionizing radiation, i.e., gamma rays, is becoming more widespread every year. The advantages of gamma radiation with composite materials are numerous; this includes continuous operation, curing at ambient temperatures, less atmospheric pollution, minimum time requirement, increased design flexibility through process control [23]. The present investigation aims at evaluating the application of gamma-irradiation doses on the physicomechanical properties of both untreated and treated particle board composites with silane coupling agent as well as the effect of the incorporation of various nanoclay contents on the properties of the prepared composites.

Experimental

Materials

Rice husk (RH) fibers were washed with water to remove residues and then dried for 24 h at 75°C before being used. They were, then, sieved to obtain particle size in the range of 0.5-0.8 mm. The polymer used in this study was epoxy resin (KEMAPOXY 150), supplied by Modern Building Chemicals Company (MBC), Egypt. It was a transparent liquid with a density of 1.8 g/cm³, and its chemical structure was presented in scheme 1.



The nanoclay (NC) was obtained from National Cement Company, Egypt. It was prepared by thermal activation of kaolin clay for 2 hours at 750°C to give active amorphous nanoclay and its chemical composition is given in Table (1). The particle size of NC was around 65.4 nm and it was measured using Transmission electron microscopy (TEM) as shown in Fig. (1). The coupling agent used in this study was 3-aminopropyl trimethoxysilane supplied by Sigma-Aldrich Company, with a chemical formula $H_2N(CH_2)_3Si(OCH_3)_3$. It was a colorless liquid, 97% concentration, and its density was 1.027 g/cm³.

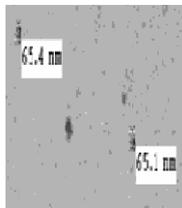


Fig.1. Transmission electron microscopy of nanoclay.

Treatment of RH fibers with silane coupling agent

The coupling agent, 3-aminopropyltrimethoxysilane was delivered in a liquid form, the amount of coupling agent applied was 2% by the weight of rice husk (RH) fibers.

Before application, it was diluted with a mixture of methanol/distilled water (80/20) and acetic acid was used to adjust pH value at 4. The fibers were soaked in this solution for 2 hours and then dried in a drying oven at 70°C.

Preparation of particle board composites (PBC)

In this part both untreated and treated rice husk fibers (RH) were mixed with epoxy resin (EP) in a ratio of 70% RH: 30%EP. Other mixtures were prepared with a partial replacement of epoxy polymer with different ratios of nanoclay 5%, 10%, 15% and 20% by the weight of the polymer to obtain particleboard composites with mix compositions designated as shown in Table (2). All mixtures were pressed into a mould of dimensions 16 x 16 x 0.8 cm, using an electric hot press type Carver-M-154. Hot pressing was performed at 120°C. Pre-heating for 5 minutes at 20000 PSI was carried out and then, the specimens were cooled at the same pressure for other 5 minutes. In addition, two other groups of untreated and treated particleboard composites with the mix composition 70% RH:(20%EP+10%NC) were prepared under the same previous conditions and subjected to different doses of gamma-irradiation (5, 10, 15, 30, 50 and 70 kGy). In the present work, the irradiation process to the required doses was carried out by gamma rays of Co-60 source using a Gamma cell type 4000A.

Table (1) Chemical oxide composition of nanoclay

Filler type	Chemical oxide composition (%)								
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	TiO ₂	K ₂ O	MnO
Nanoclay	61.5	19.8	6.3	5.7	-	-	3.4	1.9	-
L.O.I	1.3								

Table (2) Mix proportions of different particleboard composites

Polymer type	Nanoclay %	Mix composition
Epoxy resin (EP)	0	70%RH:30%EP
	5	70%RH:(25%EP+5%NC)
	10	70%RH:(20%EP+10%NC)
	15	70%RH:(15%EP+15%NC)
	20	70%RH:(10%EP+20%NC)

Physico-mechanical measurements

Flexural and impact strengths

Flexural and impact strengths are measured to evaluate the mechanical behavior according to the ASTM standards (D1037-11, 1987) [24] and (D 256, 1987) [25] respectively as shown in the following equations.

$$\text{Flexural strength} = \frac{3FL}{2bd^2} \quad (1)$$

Where,

the b = width of the specimen in cm,

d = thickness of specimen in cm,

L = length of the specimen in cm, and

F = maximum load.

$$\text{Impact strength} = \frac{A_n}{b.h} \quad \text{J/cm}^2 \quad (2)$$

Where, the A_n = absorbed impact energy in joule,

b = width of the sample in its center in cm, and

h = height of the sample in its center in cm.

Water absorption

Also, water absorption percentage as a physical property is measured according to ASTM (D1037-100, 1987) as follows:

$$\text{Water absorption (\%)} = \frac{W_2 - W_1}{W_1} \times 100 \quad (3)$$

Where, W_2 = refers to the sample weight after immersion in water, and

W_1 = represent the original weight of the sample before immersion in water.

Thermogravimetric analysis (TGA)

The thermal behavior of the specimens was determined using thermogravimetric analyzer model Shimadzu TGA-50 (Kyoto, Japan), with a flow rate of nitrogen gas of 20ml/min and a heating rate of 10°C/min.

Scanning electron microscopy (SEM)

The morphology of the fractured surface of some selected samples was investigated using scanning electron microscopy (SEM). The SEM micrographs were taken using a JSM.5400 (JEOL/Japan).

Results and discussion

Flexural strength

The influence of nanoclay content on the flexural strength values of both untreated and treated rice husk-epoxy particleboard composites is given in Fig. (2). In general, the incorporation of nanoclay particles as a partial substitution of the epoxy polymer into rice husk-epoxy particleboard composites leads to an enhancement in the flexural strength values as compared to the control particleboard composites that containing zero nanoclay content 70% RH: 30% EP. The use of nanoclay particles as a reinforcement in the rice husk-epoxy particleboard composites has significantly improved the flexural properties through the existence of interlocking and bridging effects [26]. From the figure, it is clear that the flexural strength values of both the untreated and treated particleboard composites increase with increasing nanoclay content up to a certain value (10%NC), their values are about 98 MPa, and 115 MPa respectively and then decrease. The data also indicate that the relative increase in the flexural strength values of both the untreated and treated particleboard composites that containing 10% NC is approximately about 9% and 15% as compared to the values of their control particleboard composites that contain zero content of NC. This enhancement in the flexural strength, up to 10%NC, may be attributed to the fact that with a lower loading of nanoclay, the potential of the formation of micro-voids is less, and the dispersion is more uniform which leads to a strength improvement. This is in addition to a good intermolecular attraction force (physical adsorption) between nanoclay particles and the OH groups of rice husk fiber that may also occur. Whereas, at higher ratios of nanoclay (15% and 20%), the flexural strength values are (95 MPa, and 92.4 MPa) and (109 MPa and 106 MPa) for the untreated and treated specimens respectively with an increment of about (5.5%, and 2.7%) and (9%, and 6%) compared to the control specimens containing 0% of nanoclay. This relatively low increase in strength values with 15% and 20% NC can be attributed to the poor dispersion of the nanoparticles inside the polymer forming agglomerations which act as stress concentrators leading to a reduction in flexural strength. Moreover, as nanoclay content exceeds a certain limit, small air-bubbles will be trapped in the polymer during mixing process leading to the formation of tiny voids in the composite.

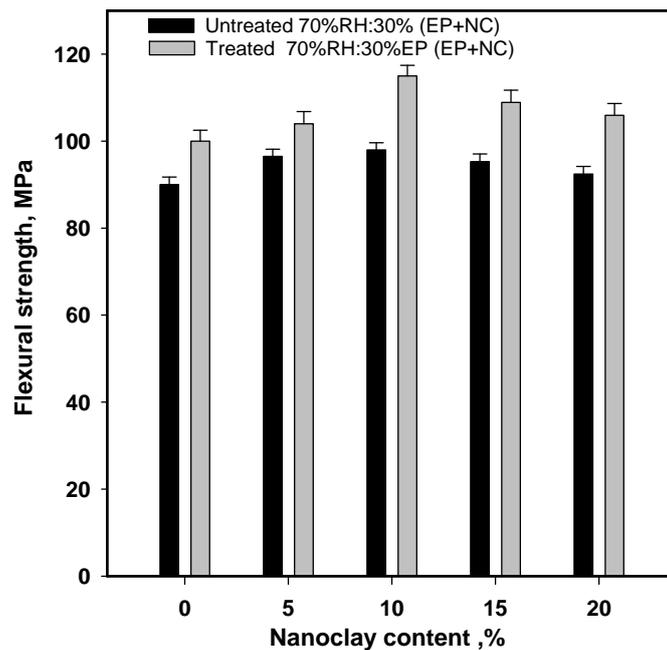
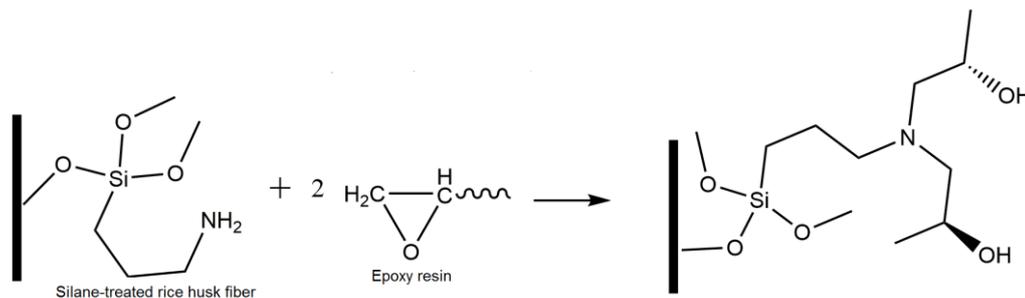


Fig.2. Flexural strength of both untreated and treated particleboard composites containing different ratios of nanoclay.



Scheme 2. Interaction mechanism of treated rice husk fiber and epoxy polymer.

On the other hand, it is also observed that the treated particleboard composites have higher flexural strength values as compared to the untreated particleboard composites having the same ratios of nanoclay. The improvement in the mechanical properties of the treated composites with 3-aminopropyl trimethoxysilane as compared to the untreated ones has been proposed to be due to the strong interaction of silane coupling agent with natural fiber either by the reaction of OH group of silanol with cellulose or by the condensation of the free silanol groups forming a rigid polysiloxane structures linked with a stable Si-O-Si- bond [27-29]. The coupling mechanism of aminosilanes in the natural fiber/epoxy composites has been summarized as in scheme 2. Also, the high reactivity of epoxy polymer toward the amino group of silane coupling agent [30], in addition to the improved interfacial interaction between nanoclay oxides and epoxy polymer are responsible for the improvement of the flexural strength of the treated composites.

The effect of different gamma-irradiation doses on the flexural strength values of both the untreated and treated rice husk-epoxy particleboard composites containing 10% of nanoclay is shown in Fig. (3). The results show that the flexural strength for both the untreated and treated particleboard composites having a mix composition 70% RH: (20% EP+10% NC) has maximum values 108 MPa, and 123 MPa at a dose of 10 kGy respectively and then, slightly decreases with increasing the irradiation dose up to 70 kGy. Among the advantages of ionizing radiation is its high penetration, which enables it to irradiate bulky products with large volumes or odd shapes. Besides, crosslinking and chain scission are two competing reactions induced by ionizing radiation of polymers and can happen simultaneously. Consequently, the improvement in the flexural strength at a dose of 10 kGy may be due to the fact that when the polymeric resin is irradiated, free

radicals are formed and cross-linking mechanism can occur. While with increasing gamma-irradiation dose beyond 10 kGy, the flexural strength values slightly decrease for all particleboard composites due to some chain sessions of the polymer. This is in addition to the degradation of the cellulosic fiber when exposed to higher doses of ionizing radiation. The results also indicated that for any given gamma-irradiation dose the flexural strength values of the treated rice husk-epoxy composite with silane coupling agent are higher than those of the untreated ones. This may be due to the presence of silane coupling agent which also participates in the crosslinking process of epoxy polymer and acts as a good chemical bridge that connects the organic polymer with nanoclay and binds them strongly.

Impact strength

The impact properties of the particleboard composites depend mainly on the properties of the individual fibers used, inter-laminar and interfacial adhesion between the fiber and the polymer [31]. Figure (4) shows the effect of different ratios of nanoclay on the impact strength values of both untreated and treated particleboard composites. From this figure, the results evidently show that the incorporation of nanoclay particles up to 10% improves the impact strength properties primarily which may be due to the good interfacial interaction between the polymer and nanoclay. Also, this could be attributed to the high aspect ratio of the dispersed particles, which results in a relatively a smaller number of cracks and better interfacial bonding in the particleboard composites as compared to the control particleboard composite i.e., containing zero % of nanoclay. Moreover, the enhanced impact strength properties may be attributed to the high stiffness of nanoclay-reinforced particleboard composites, which occurs due to the reduced mobility of polymer that is intercalated between interlayers [32]. As a result of the abovementioned, the absorption of applied energy becomes easier which prevents the crack initiation and its propagation within

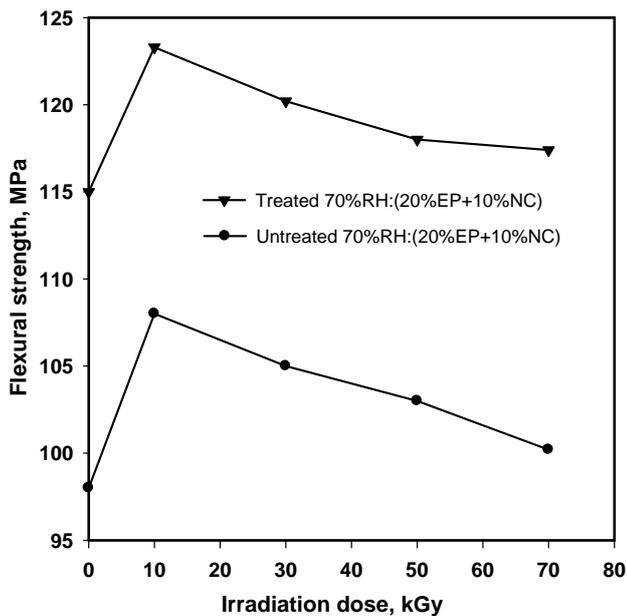


Fig.3. Effect of different gamma-irradiation doses on flexural strength values of particleboard composites.

the composite materials under stress load. However, at higher loadings of nanoclay (15% and 20%) a reduction in the values of impact strength for all untreated and treated particleboard composites takes place. This behavior may be due to the stress concentration effect of agglomerated nanoparticles. This agglomeration leads to a reduction in mechanical properties due to lowering nanoclay surface area and as a result, a lower polymer/filler surface interaction takes place. In addition, at any given nanoclay content, it is clearly observed that the impact strength values of the treated particleboard composites with 3-aminopropyltrimethoxysilane have better values compared to the untreated ones. This is due to the role of the amino silane coupling agent which facilitates and increases the interfacial adhesion between hydrophilic rice husk fiber and the hydrophobic epoxy resin and as a result, the ability of the specimens to withstand a suddenly applied load is increased.

The influence of different gamma-irradiation doses on the impact strength of particleboard composites containing 10% nanoclay is graphically presented in Fig. (5). The results revealed that the impact strength has a maximum value of 10 kGy. This may be attributed to the interaction between hydroxyl groups presented in the RH fibers surface, the active polymer species, and nanoclay particles during the gamma-irradiation process. While at higher gamma-irradiation doses, a reduction of the impact strength occurs due to the degradation of polymer and fiber.

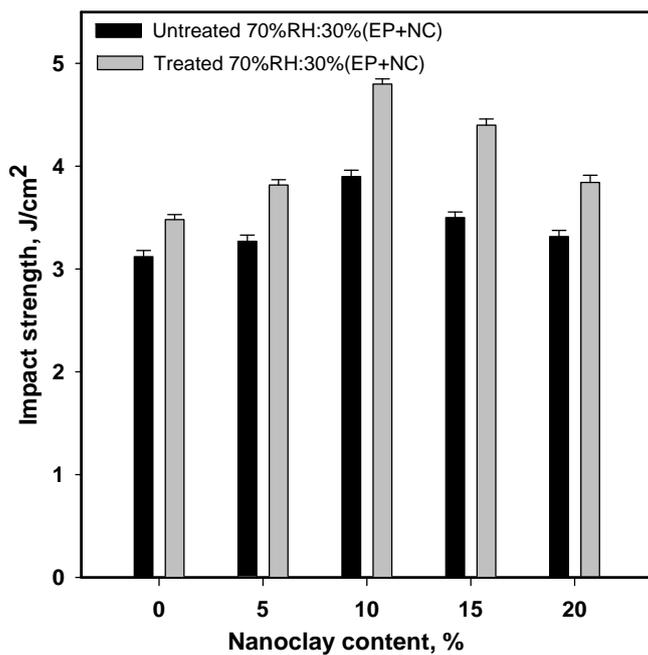


Fig. 4. Impact strength of both untreated and treated particleboard composites containing different ratios of nanoclay.

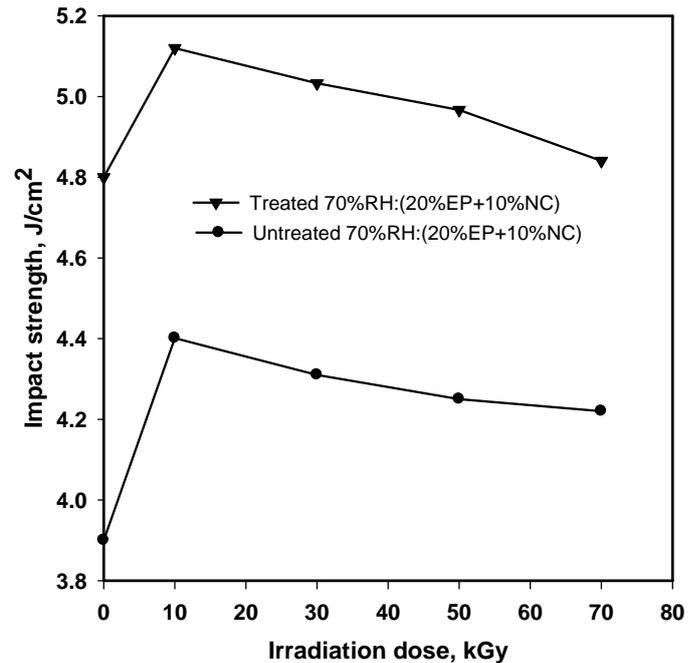


Fig.5. Effect of different gamma-irradiation doses on impact strength values of particleboard composites.

Water absorption (%)

The effect of nanoclay content on the water absorption (%) of the untreated and treated particleboard composites is presented in Fig. (6). It is clearly observed that the incorporation of nanoclay effectively decreases the water absorption percentages for particleboard composites that contain nanoclay as compared to control particleboard composites 70% RH:30% EP (zero% NC). The water absorption (%) decreases continuously with increasing nanoclay contents up to 10% for both the untreated and treated particleboard composite specimens and then starts to increase at higher loadings of nanoclay up to 20%, but it is still lower as compared to the control specimens. This behavior can be attributed to the presence of nanoparticles which acts as a barrier medium that hinders the water flow into the composites from all direction, thus resulting in a decreased equilibrium water content as reported in the literature [33-35]. For more clarification, the incorporation of a small amount of nanoparticles usually increases the barrier properties of

nanocomposites against oxygen, nitrogen, carbon dioxide, water vapor, and reduces the water uptake [36-38].

The effect of γ -irradiation doses on the water absorption percentages of the untreated and treated particleboard composites containing 10%NC is given in Fig. (7). The results show that the water absorption values for all specimens decrease at a dose of 10 kGy of gamma-irradiation and then increase for all particleboard composites. In addition, it is also observed that the values of water absorption percentages of the treated particleboard composite are lower as compared to those made with the untreated fibers. In other words, it is perceived from the figure that at a lower gamma radiation dose (10 kGy), the value of water absorption (%) shows an improvement (decreases) due to cross-linking and hence better fiber polymer adhesion takes place. While at higher doses of radiation, the values of water absorption (%) increase due to the degradation of the polymeric molecules.

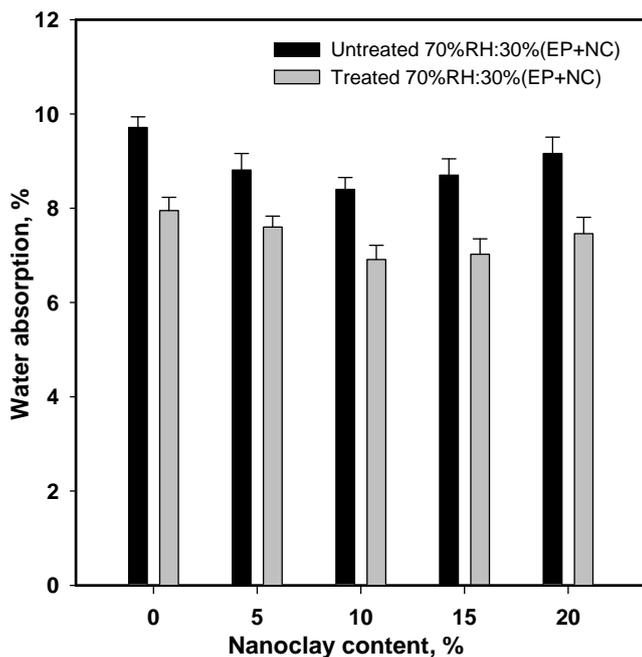


Fig.6. Water absorption percentages of both untreated and treated particleboard composites containing different ratios of nanoclay.

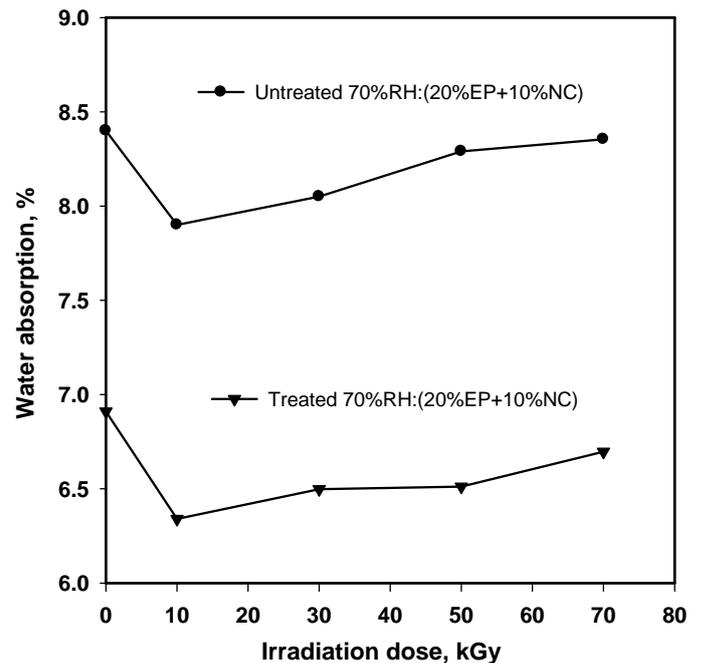


Fig.7. Effect of different gamma-irradiation doses on water absorption percentages of particleboard composites.

Thermogravimetric analysis (TGA)

The thermogravimetric analysis was used to investigate the thermal behavior of both the untreated and treated particleboard composites having a mix composition 70% RH: (20% EP+10% NC) before and after they had irradiated with a dose of 10 kGy as shown in Fig. (8). The TGA thermogram was used to describe the variation of weight remaining percentages as a measure of the weight loss with a progressive temperature raise from room temperature up to a maximum temperature of 900°C at a heating rate of 10°C/min. The general thermal behavior of the specimens showed that the weight remaining percentages of both the untreated and treated particleboard composites at 0 kGy and 10 kGy of gamma-irradiation are slightly decreased in the temperature range 100–200°C. This behaviour may be related to the loss of physically absorbed water in the rice husk fibers, loss of light volatiles, and breaking of unreacted epoxy or other traces of impurities. Above this temperature, the TGA curve exhibited two stages, a gradual decrease in the weight remaining percentages which corresponded to the thermal degradation of hemicellulose of rice husk fiber within the temperature range 200–320°C. In the second stage, the degradation started at 330–550°C due to chain scission of the epoxy

polymer and then started leveling off in a plateau at 600°C

Table (3) summarizes the weight remaining percentages at different temperatures taken from the TGA thermograms. Comparing the data obtained from the TGA thermal curve Fig. (9) and Table (3), it was clear that the order of the values of weight remaining percentages at a high temperature of 900°C was ranked as follows: treated particleboard irradiated at 10 kGy > treated particleboard at 0 kGy > untreated particleboard irradiated at 10 kGy > untreated particleboard at 0 kGy, their values were 26.2, 23.1, 20.7, and 14.1 respectively. From the above, it was clear that the treated particleboard specimens that irradiated at 10 kGy were thermally more stable as compared to the specimens of other composites and this was consistent with the mechanical results that have been obtained by the authors. This behavior can be attributed to the fact that silane possesses functional active end groups, which lead to an improvement in the rice husk–epoxy interaction [39].

On the other hand, the rate of thermal decomposition reaction dw/dt (taken from the initial TGA thermograms) of both the untreated and treated particleboard composites 70% RH: (20% EP+10% NC) before and after irradiation

with gamma rays was plotted as a function of temperature as shown in Fig. (9). The results showed that the maximum value of the reaction rate of thermal decomposition is shifted towards higher temperatures for the treated particleboard composites irradiated at a dose of 10 kGy. The $T_{max}^{\circ}C$ was located at temperatures 312 $^{\circ}C$ and 328 $^{\circ}C$ for the treated particleboard composites at zero and 10kGy respectively. While the $T_{max}^{\circ}C$ values for the untreated composites whether irradiated or not were located at 302 $^{\circ}C$.

Scanning electron microscope (SEM)

Figure (10) shows the SEM micrographs of the fractured surfaces of the treated rice husk/epoxy particleboard composite (70% RH: 30% EP),

treated particleboard composites containing 10% of nanoclay 70% RH: (20% EP+10% NC) before and after they had been irradiated at a dose of 10 kGy respectively. It can be seen from the first image (a), that the absence of nanoclay leads to the appearance of some spaces and cavities between the constituents of the particleboard composites while in the presence of 10% nanoclay in the second (b) and third (c) images a dense structure is formed with less cavities specially in the third micrograph which is under the effect of a dose of 10 kGy of gamma irradiation leading to a crosslinked polymer and a network structure that surrounded the fibers to a large extent and created a proper adhesion.

Table (3) Weight remaining % of the untreated and treated particleboard composites containing 10% nanoclay at 0kGy and 10kGy

Temperature (°C)	Weight remaining (%) of particleboard composites			
	Treated at 10kGy	Treated at 0kGy	Untreated at 10kGy	Untreated at 0kGy
100°C	97.7	97.7	96.7	97.4
200°C	95.9	95.7	94.4	96.4
300°C	74.8	74.8	83.6	79.1
400°C	47.6	44.5	44.4	42.1
500°C	31.7	27.3	26.5	29.5
600°C	25.3	21.3	19.3	13.9
700°C	25.4	21.7	19.6	13.7
800°C	26.1	22.4	20.0	13.9
900°C	26.2	23.1	20.7	14.2

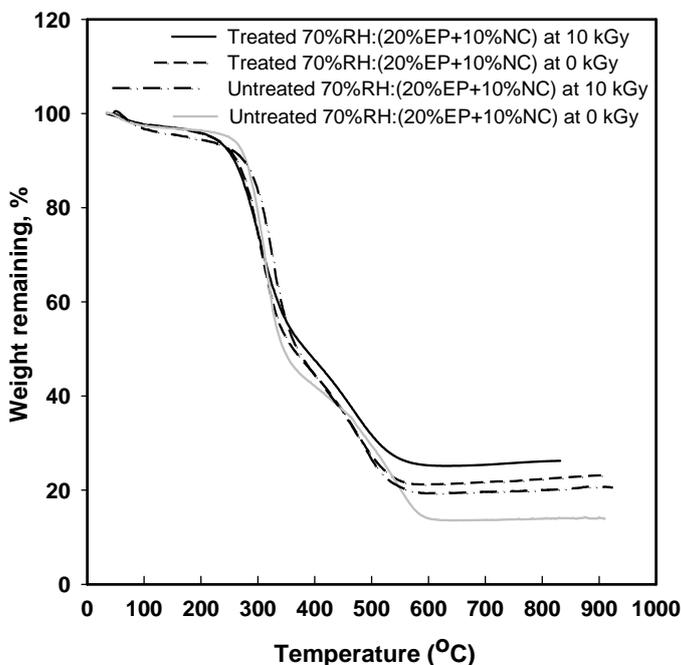


Fig.8. TGA thermograms of both untreated and treated particleboard composites containing 10% nanoclay at 0 and 10kGy of gamma rays.

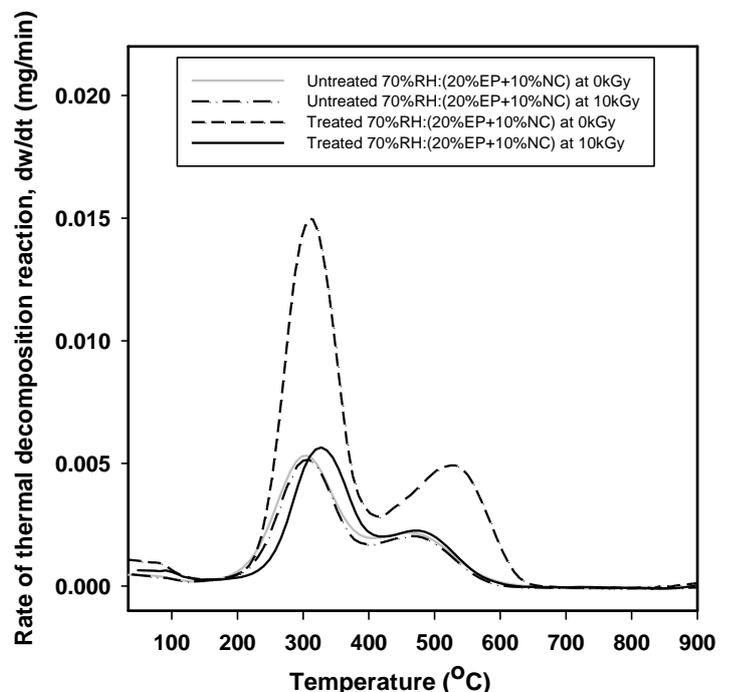


Fig.9. Rate of thermal decomposition reaction (dw/dt) of both untreated and treated particleboard composites containing 10% nanoclay at 0kGy and 10 kGy of gamma rays.

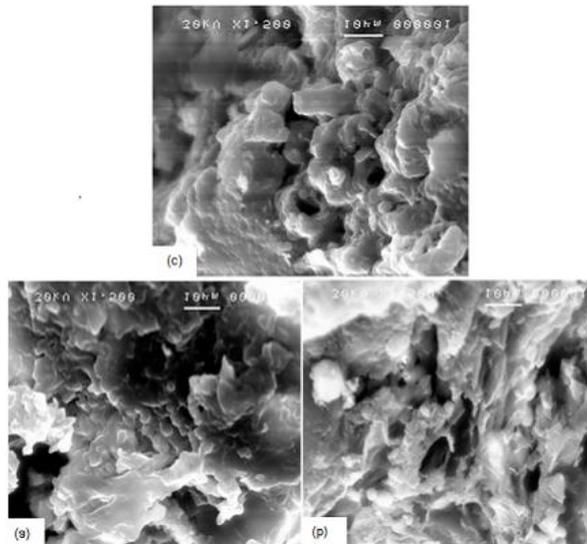


Fig.10. SEM micrographs for (a) treated rice husk /epoxy particleboard composite (70%RH:30%EP), (b) treated particleboard composite containing 10%NC at 0kGy, and (c) treated particleboard composite containing 10%NC at a dose of 10kGy.

Conclusion

The treated particleboard composites with 3-aminopropyltrimethoxy silane exhibited improved properties as compared to untreated composites prepared under the same conditions. The use of 10% nanoclay has been an attractive approach to enhance the physico-mechanical properties of both the untreated and treated rice husk-epoxy particleboard composites.

TGA thermograms indicated that the treated particleboard composite is thermally more stable than the untreated ones. The irradiated specimens at a dose of 10 kGy give good physico-mechanical and thermal properties as compared to the unirradiated composite.

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