Mechanical properties of nanocomposites for building materials
R M Salih* and Sh H Ahmad
Department of Applied Sciences, University of Technology, Baghdad, Iraq
*Corresponding author email: ranamahdi1@gmail.com

Abstract

This work aims to evaluate some mechanical properties (bending behavior, compressive strength and impact strength) for polymeric composites made of a polymer blend (epoxy+ polyester) as a matrix, with a hybrid reinforcement of both rock wool and nano clay. Hand lay-up technique was used to manufacture the castings, and epoxy and polyester were mixed in different weight ratios, starting from (50:50) up to (90:10) % of epoxy and polyester respectively. Optimum mixing ratio (OMR) was decided upon carrying out impact test on the specimens, and was then reinforced with nanoclay (Kaolite) in weight fractions (5 and 7.5)%.. These ratios were then hybridized with rockwool, added in a volume fraction (10%). Mechanical properties (bending behavior, compressive strength, and impact strength) were studied for these specimens. The results show that the blend reinforced with nano clay in a weight fraction (7.5)% and hybridized with rockwool volume fraction (10%) had the lowest deflection and the highest value of Young’s modulus (1889.49)MPa in bending, while blend reinforced with 5% nanoclay and rockwool (10%) showed the highest value of compressive strength (396.95) MPa. The reinforcement with nanoclay reduce the impact strength, the lowest impact strength was (0.0055)KJ/m² for (blend+5% nanoclay).

Key Words: Mechanical properties, Nano clays, Nanocomposite, Polymer blends.

1. Introduction

Polymer nanocomposites have attracted great interest in recent years because of their remarkable improvement of physical, mechanical and thermal properties at low loadings of fillers. This enhancement is due to the unique characteristics of the nanometer-sized filler particles such as large surface to volume ratio of the filler which highly improves the
properties of the nanocomposites. Nanostructures have gained much attention since the properties of nanocrystals depend not only on their composition but also on their shape, size, structure, phase, and size distribution [1]. Important factors that can affect the physical and mechanical properties of nano-filler-reinforced polymer nanocomposites include nano-filler type, filler loading levels, and processing. Nanoclay, an inexpensive natural mineral, has been reported by many studies as one of the potential candidates for nanocomposites [2].

Many of the previous instances are nanocomposite materials; this is, they are the combination of traditional materials such as ceramics, metals and polymers with nanomaterials (e.g., nanosilica, nanotubes, nanofibers, etc.) in different phases: continuous, for example nano-silica in a binding matrix (the case of concretes and mortars based on Portland cement) and disperse (with aggregates such as nanofibers, nanotubes and other reinforcements by precipitation) which, when added, would yield nanocomposite materials to improve many of the physical and chemical properties that composite materials normally have [3].

Ahmad Rafiq and Necar Merah studied glass fiber-reinforced epoxy-nanoclay composite plates, with I.30E clay contents ranging between 0 and 5 wt.%, were manufactured by hand layup with hot pressing. Flexural strength of unexposed fiber-reinforced epoxy-nanoclay reached an optimum improvement of 11% for 1.5 wt.%. Scanning electron microscope analysis showed that at this clay loading, better interfacial adhesion of clay with glass fibers was achieved. At higher clay loadings, clay agglomeration and presence of micro-voids led to less strength improvement. The maximum water uptake was found to decrease with increasing clay loading and moisture diffusion at 80°C Exposure to moisture resulted in degradation of fiber-reinforced epoxy-nanoclay flexural properties with about 36% reduction in strength for 80°C and 8% for room temperature [4].

JR Robledo-Ortiz et al. studied three different types of nanoclays (1.44P, 1.34MN, and Cloisite 15A) which were used to reinforce an injection grade poly lactic acid (PLA). The nanocomposites (NCs) were prepared using three different nanoclay concentration levels (1, 3, and 5 wt%) in a twin-screw extruder. Results showed that the three nanoclays significantly increased the tensile and flexural modulus of the injection grade PLA. The 1.34MN NCs also showed improvement in the tensile strength, in addition to improving flexural strength. Additionally, the use of 5 wt% of 1.44P nanoclay allowed an increase in impact strength while using 1.34MN and 15A nanoclays, the impact strength was similar to the one observed for pure PLA. In general, mechanodynamic analysis results showed that storage modulus increased with nanoclay content; while thermogravimetric analysis indicated that none of the nanoclays has a significant effect over the degradation temperature of pure PLA. Differential scanning calorimetry results showed that the crystallinity of PLA is enhanced with nanoclay inclusion. For 1.34MN NCs, X-ray diffraction observations exposed that the mineral clay relative intensity peaks disappeared indicating nanoclay exfoliation, which contributes to the increase in tensile and flexural strength in the NCs. Nevertheless for 1.44P and 15A nanoclays, an increase in the interlayer distance (intercalation) was detected [5].

2. Experimental work

2.1. Materials

2.1.1 Unsaturated Polyester (UP)

A Thermoset polymer that is usually transparent viscous liquid, which solidifies upon the addition of a hardener (methyl ethyl ketone peroxide-MEK P) in a weight ratio 1%. The unsaturated polyester was supplied by Saudi Industrial Resins Limited Company (SIR)™. Table (1) gives some of it’s properties.

| Table (1). General properties of polyester resin. |
2.1.2 Epoxy Resin

Epoxy resin that used in this work was Sikadur–105, manufactured by Sika™, which is a two component, low viscosity epoxy resin system in the form of transparent liquid (which transforms into solid state after adding the hardener to it in a percentage of 2:1). Table (2) how the properties of epoxy resin.

| Property               | Value                                      |
|------------------------|--------------------------------------------|
| Flexural properties    | 14 day 7900 psi(54 MPa)                    |
| Viscosity (mixed)      | 200 cps                                    |
| Pot life               | 30 minutes.(60 gm mass)                    |
| Tensile properties     | 14 day 4500 psi(37.2 MPa)                  |
| 14 day tensile strength| 54 MPa                                     |
| Elongation at break    | 3.1%                                       |
| Modulus of elasticity  | 1400 MPa                                    |

Table (2). General properties of epoxy resin.

2.1.3 Nanoclay (Kaolinite)

Kaolinite is known for its abundance and desired properties, leading to its use in a wide range of plastics, cements and catalysts. The nano clay used in this study was provided by Intelligent Materials Pvt. Ltd.™, properties are shown in table (3).

| Property       | Value                        |
|----------------|------------------------------|
| Molecular weight | 258.2 g/mol.                |
| Appearance     | White powder                 |
| Specific gravity | ~2.6                        |
| Melting point  | >1500 °C                     |
| Solubility     | Insoluble in cold water      |

Table (3). General properties of nanoclay.

2.1.4 Rockwool:

Sometimes called stone wool, is a type of inorganic fiber. The loose rockwool used in this study was manufactured by Saudi Rockwool™. It is primarily used for thermal and acoustical insulation, typically in buildings, vehicles and other industrial equipments. It has a high tensile strength and modulus, high chemical resistance, high dimensional stability, and has excellent insulation properties. The table (4) shows the general properties of rockwool.

| Property            | Value                                      |
|---------------------|--------------------------------------------|
| Designated use      | Loose filler for heaters e, ovens, cookers, and for brake pads |
| Desired properties  | Reduction of transferred heat, and transmitted noise. |
| Appearance          | Mixed black and yellow                    |
| Packing             | Loose wool supplied in PE bags             |

Table (4). General properties of rockwool.
2.2. Method

Hand lay-up method was used to prepare samples with mold size of (250mm*250mm). Unsaturated polyester was mixed with epoxy to form a blend. Five different percentages of epoxy and polyester were mixed, starting from (90:10)%, (80:20)%, (70:30)%, (60:40)%, of epoxy and polyester respectively, and reaching (50:50)% of both. The optimum mixing ratio (OMR) was decided upon the results of the impact test carried out on blends of the above mentioned percentages. The (80:20)% has shown the highest impact strength value, hence was selected as a matrix to the composite, which was reinforced with 2 weight fractions: (5)% and (7.5)% of nano clay, then rockwool was added to prepare two other castings with a volume fraction(10%), hybridized with the previously mentioned weight fractions of nano clay. A single casting was made of solely epoxy/polyester blend for the purpose of comparison.

All castings were left for 24 hours for primary solidification, later they were taken out of the mold and were put in a dryer for curing and stress removal. The castings are shown in figure. (1) below.

![Figure (1). The samples](image)

2.3. The tests

2.3.1 Bending Test (3-Point test)

This test was carried out using the bending set (PHYWE) shown in figure (2), in compliance with the standard specification (ASTM -790). Young’s modulus values for the specimens was calculated using the following formula:

\[ E = \frac{MgL^3}{48IS} \] ...

(1)

Where M: mass (gm), g: Earth’s gravitation (9.8 m/s²), L: distance between the two supports (mm), S: deflection (mm), I: geometrical bending moment (mm⁴), and the latter can be calculated using the following equation:

\[ I = \frac{bd^3}{12} \] ...

(2)

4
2.3.2 Compression Test
Compression test was done using a “Leybold-Harris” hydraulic press, shown in figure (3), in accordance with standard specification (ASTM D-695). The value of compressive strength (UCS) was gained by dividing the maximum load by the cross sectional area of the specimens, and it gives the value of compressive load under which the material fails.

\[ I.S. = \frac{U_s}{A} \]  

Where, I.S.: the impact strength (kJ/m$^2$), Us: the fracture energy (kJ) and A: is the cross sectional area of the sample (m$^2$). Fig. (4) shows the impact test samples.
3. Results and discussion

3.1 Bending test

From the bending test results, the highest deflection rate was noticed for the unreinforced blend specimen, and the deflection decreased when the blend was reinforced with (5)% and (7.5)%, and the value of Young’s modulus reached (746.8) MPa when nanoclay is (7.5)%, Then when hybridized with rockwool (10%) volume fraction, the Young’s modulus has further increased significantly to (1887.49) MPa, as show in figure (5).

Instead they tended to break apart before the peak, which meant they were brittle. It was because when the nanoclay weight percentage increased, the mixture itself became too viscous, sluggish and more void took place in the samples of high weight fraction. The more the nanoclay added, the more viscous of the clay–resin mixture, leading to a difficulty in even and equal distribution of nanoclay over the whole mixture prior to casting (while still in liquid state). This is the reason for which the higher wt% samples failed. Another reason for failure of higher clay loading is low aspect ratio of clay particles and low contact surface area resulting in weak adhesion between polymer matrix and clay.

Furthermore, with high clay loading this behavior was probably attributed to the filler-filler interaction which resulted in agglomerates, induced local stress concentration, and finally reduced mechanical properties of the nanocomposites[6].

![Figure (4). Impact test set](image)

![Figure (5). The effect of nanoclay and rockwool on the Young's modulus determined by bending test](image)
3.2 Compressive test

Figure (6) show the effect of (5)% and (7.5)% weight fraction of nanoclay on the compressive strength. It was found that the nanoclay enhanced the compressive modulus of the (epoxy/polyester) blend. However a detrimental effect on the compressive yield strength and failure strain was observed. This shows that the intercalated structure of nanoclay create high localized stresses in the matrix during compression. The results show that the compressive strength for (polyester/epoxy) blend is 210.68 MPa. A slight increase in this value occurred when nanoclay was added with a ratio (5%) weight fraction. The highest value of compressive strength was observed with the hybrid reinforcement of (5%) nanoclay + (10%) rockwool, to be 396.95 MPa. While reinforcement with (7.5%) weight fraction nanoclay decreased compressive strength of the resulting composite[7]. the compressive strength increased with an increase of the RW(rockwool). The rockwool in the composites acted as a pozzolanic material, giving rise to good mechanical properties, including compressive strength, due to a higher density of the final product. In addition, the specimens containing (10)% rockwool had higher compressive strength that those without rockwool. This might be due to the particle packing effect by the finer RW, which can fill voids [8].

![compressive strength graph](image)

**Figure(6).** the effect of nanoclay and rockwool on compressive strength

3.3 Impact test

In this test the result show that the highest impact strength was noticed in pure specimen (epoxy/polyester) blend, with a value (0.017) KJ/m², the impact strength begin to reduce significantly when reinforce the blend with nanoclay (5%,7.5%) and hybrid with rockwool (10%) volume fraction reached lowest impact strength (0.0055) when the ratio of nanoclay is (5%) weight fraction.

There was a sharp drop of nearly 11.5% in impact strength when (5)% wt.clay was added into the blend, followed by little variation with further increase in the clay content Figure (7).

The failure mechanisms in the impact specimens were somehow different from those of the bending fracture specimens, the specimen with added nanoclay became brittle under the high strain rate loading, leading to nano clay particles acting as stress concentrators in the matrix, which can fracture easily due to the formation of microvoids even before the cracks reached the surrounding matrix material[9]. The fracture in the specimen which contain rockwool fiber is brittle because the rockwool is basically a basalt rocks which brittleness is one of its important characteristics[10].
3.4 SEM images of the nano composite

SEM technique was used to reveal the microscopic details of the specimens. Figures (8) shows unreinforced blend, with no clear boundaries between the two constituents (epoxy+unsaturated polyester), even with a magnification down to 50 µm. The image shows a breaking lines along the surface fractography, indicating a brittle fractured surface, along with some voids scattered within the whole volume of the specimen.

Figures (8). The effect of nano clay and rockwool on the impact strength
Figures (9), show the blend reinforced with 5% wt. nano clay (left) and the blend reinforced with 5% wt. nano clay and 10% vol. rockwool (right).

Figures (9) on the left shows the blend reinforced with 5% nano clay as a reinforcement, in this specimen nano clay is agglomerated in small clusters that are scattered throughout the matrix, leaving free space between them (which is free of nano clay). These agglomerates emphasize the nature of nano additions in general, such that even with the use of ultrasonication, the nano clays tend to form clusters, dispersed throughout the matrix. The situation changed a bit after the addition of 10% wt. fraction rockwool to the (5%) nano clay reinforced specimen. Figure (9) on the right, the rockwool fibers are visible even with a relatively low magnification power (100µm), the fibers are used in a random mat shape, which explains the random nature of their dispersion in the matrix (no regular pattern is noticed whether at the macro-, or micro- scale), and the micrographs show small or no plastic deformation to the fractured fibers, since no change in dimensions or necking took place along the fibers.

Figures (10), show the blend reinforced with 7.5% wt. nano clay (left) and blend reinforced with 7.5% wt. nanoclay and 10% vol. rockwool (right).

Blend reinforced with 7.5% wt. nanoclay is shown in figures (10) on the left, as with the 5% nano clay reinforced specimen, the 7.5% specimen shows agglomerations of nano clay, co-existing with nano clay-free space in the matrix, with a denser packing of the agglomerates
and less free space than that observed in the 5% wt. nano clay specimen. The same figure on the right shows the blend reinforced with 7.5% wt. nano clay and 10% rockwool. Here the surface fractography shows the brittle nature of fracture. Here the fiber cross section is smooth and no necking/ or change in the dimensions of the fibers occurred, beside the flat lines shown on the surface of the nano clay-free volume. Again the nano clays is agglomerated in small gatherings which are scattered though the matrix.

4. Conclusion.
Epoxy and polyester can be mixed in different percentages without visible phase segregation, however, optimum mixing ratio can be decided upon carrying out the impact test, through which the specimen giving the highest value can be chosen to work with. Through the results the highest impact strength was found in the unreinforced blend and the lowest value was for the 5% nanoclay reinforced blend. The result of bending strength proved that the highest deflection was seen in unreinforced blend and the lowest deflection was when reinforced with nanoclay 7.5%wt and rockwool 10% vol fraction. In compressive test the results showed that the addition of nano clay 5% weight fraction, hybridized with 10% vol. fraction rockwool, increased the compressive strength. SEM images showed that there is no interface between the matrix phases so they couldn’t be distinguished apart. The surface of fracture was brittle with existence of scattered voids, nano agglomeration and nanoclay-free space.

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