Preparation and investigation of physical and mechanical properties of vinyl ester/graphene oxide nanocomposite.

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Abstract

Composite materials can be used as coatings in various industries, including chemical and water industries, to prevent damage and corrosion of equipment. Nano-reinforcements, due to their very small dimensions and high surface area compared to conventional reinforcements, improve the desired properties and are a good substitute for conventional composites. Considering the mentioned factors, choosing a suitable nano-composite system for use as a resistant and strong coating in equipment is necessary, which can reduce the operating costs of companies. In this thesis, the physical, mechanical, and wear properties of vinyl ester nano-composites reinforced with graphene oxide have been investigated. First, three different samples of vinyl ester nano-composites containing 0.5%, 1.0%, and 2.0% by weight of graphene oxide/vinyl ester composites were prepared and fabricated. Then, the properties of the samples were obtained using tensile, reactivity, XRD, and SEM tests. The results showed that the optimum amount of graphene oxide nanoparticles in the weight percentage of 0.1% compared to vinyl ester resin was 151.9 degrees Celsius in the reactivity test and 87.06 megapascals in the tensile strength test.

Key words: *Vinyl ester resin, graphene oxide, physical and mechanical properties, nanocomposite.*

Introduction:

Nanotechnology is the ability to produce and construct new materials, tools, and systems by controlling atomic and molecular levels, which exhibit properties at these levels. A nanometer is equal to one billionth of a meter (10^-9 meters). This size is 18,000 times smaller than the diameter of a human hair. On average, 3 to 6 atoms side by side make up a length equivalent to one nanometer, which depends on the type of atom. In general, nanotechnology is the development, production, and use of tools and materials with dimensions of approximately 1-100 nanometers. Increasing the ratio of surface area to volume, which gradually occurs with decreasing particle size, causes the behavior of atoms on the surface of the particle to dominate over the behavior of internal atoms [1].

In the usual case, when powder reinforcement is added to polymers, there is not much interaction between the reinforcement and the polymer. However, if the reinforcement particles become smaller, this interaction increases, and if the size of these particles is at the atomic or molecular scale, this reinforcement property reaches its maximum level. In general, reinforcement materials that have one of their dimensions in the nanoscale are called nanoreinforcements. When using these reinforcements, the resulting composite is called a nanocomposite [2].

The most important feature of nanocomposites is the improvement of properties with small amounts of reinforcement, to the extent that adding 5% by volume of nanoparticles to the polymer improves its physical-mechanical properties by up to 70%. One of the nanoreinforcements is graphene, which has been used to reinforce thermoplastic plastics such as polyolefins, nylon 6, polystyrene, and also thermosetting plastics such as epoxy [3].

An overview of the work done

Kojima and colleagues demonstrated that at values of 2.4% of nano clay particles, the modulus of elasticity doubled, tensile strength increased by 50%, and the heat deflection temperature increased by 120% compared to pure polymer [4].

Method of work:

In this study, vinyl ester resin made in Iran was used as the composite matrix, dimethyl aniline made in Iran was used as the process facilitator for matrix curing, cobalt acetate was used as the accelerator for matrix curing, and methyl ethyl ketone peroxide obtained from a Dutch company was used as the curing agent for matrix curing. It should be noted that the graphene oxide used in this study was prepared as a reinforcing phase in the composite through a modified Hummers reaction.

Graphene oxide synthesis

Graphene oxide synthesis was carried out using the modified Hummers method [7]. In this method, graphite powder was used as the raw material for graphene oxide production. Initially, 3 grams of graphite (mesh500) were added to a mixture of sulfuric acid (360 milliliters), phosphoric acid (40 milliliters), and potassium permanganate (18 grams), and heated to 40 degrees Celsius. Then, the mixture was heated to 50 degrees Celsius and stirred for 12 hours. The solution was placed in an ice bath and 30 milliliters of hydrogen peroxide were added to stop the reaction. After passing through a sieve (hole size 300 micrometers) and centrifuging for 4 hours, the remaining solid materials were washed with 200 milliliters of water, 200 milliliters of 35% HCl, and 200 milliliters of ethanol. The solution was then filtered using the same method and the remaining materials were washed with 200 milliliters of ether and passed through a sieve with hole size 0.45 micrometers. Finally, the obtained powder was dried in an oven.

Preparation of Vinyl Ester/Graphene Oxide Nanocomposite

In this article, tests were conducted on 5 different samples containing weight fractions of 0.0, 0.05, 0.1, 0.2, and 0.4 of graphene oxide. Composite materials with different percentages of graphene oxide nanoparticles were prepared, and a composite with a weight fraction of 0.4% of nanoparticles was also made. Even after adding 10% curing agent, the nanocomposite did not gel within 24 hours. The vinyl ester composite, along with the supplied initiator and catalyst from Fraple Company, was mechanically mixed with the required amount of modified graphene oxide and placed in an ultrasonic bath inside an ultrasonic device. Sonication was carried out for 20 minutes at 200 watts. After complete dispersion of the nanoparticles in the resin, 1% curing agent was added to the composite sample and the sample was thoroughly mixed using a hand mixer. Then, molding was performed (Figure 1), and to remove air from the sample, it was placed under vacuum conditions. The sample was further cured using an oven for final curing.

For the vinyl ester resin, the test sample was placed in an oven at 80 degrees Celsius for 4 hours [8]. This was done until all cross-linking reactions took place and the piece was fully cured.

Making a sample from pre-prepared resin (ultrasonically treated) for tensile testing

Step 1: Weigh 200 grams of commercial grade isophthalic polyester resin, Fraple 201, using a scale with an accuracy of 0.0001/0 grams, as shown below. Then, according to the resin datasheet, add cobalt as an accelerator and P211 catalysts from the methyl ethyl ketone peroxides family as initiators and mix well.

Step 2: To make the resin sheet mold, use two glass sheets with dimensions of 20 by 25 and a plastic O-ring to be placed between the two glass sheets. Also, use four clamps to hold the glass sheets together. First, clean the surfaces of the glass sheets that will be in

contact with each other using acetone. Then, apply a very small amount of wax to facilitate the separation of the resin sheet from between the glass sheets.

Step 3: After preparing the resin mold and removing all the bubbles from it, pour the resin into the mold as shown in the figure. After 10 minutes, the resin will start to gel, and we kept it inside the mold for 24 hours until the first curing stage is complete. In this stage, the resin solidifies and hardens. This stage is also known as resin curing.

Step 4: After the first curing stage is complete, remove the cured resin from the mold. Then, according to the ISO 2-527 standard (dimensional standard for sample preparation and tensile testing), prepare the sheets using a cutting machine.

Step 5: After cutting, perform the final curing of the samples for 4 hours at a temperature of 80 degrees Celsius.

Reactivity Test

The reactivity Test

The reactivity test, determines three important factors during a chemical reaction: network formation or resin curing by the curing agent. These factors are the final heat generated during the reaction, the time it takes to reach the final heat from 25 degrees Celsius (factor b), and the time it takes for the reaction temperature to reach from 25 degrees to 35 degrees Celsius (factor a). It is usually possible to analyze the degree of resin network formation well from this test. It is worth mentioning that this test is performed based on the Flowtite R 104 standard.

Tensile Test

The tensile test is one of the destructive tests in materials science, in which a specimen is subjected to uniaxial tension until it reaches the point of fracture. The tensile specimen is a standard cross-sectional sample with two arms and a gauge section in the middle. The arms are large enough to be securely gripped by clamps, while the gauge section has a smaller cross-sectional area where deformation and fracture gradually occur. In this article, this test is conducted according to the ISO-527 standard.

Research and Conclusion

Considering the use of nanoparticles, the aim was to investigate the increase in this resistance, which in the sample with 0.05% graphene oxide nanoparticles, the amount increased by 3.12%, in the sample with 1.0% nanoparticles, it increased by 9.30%, and in the sample with 2.0% nanoparticles, it increased by 17.17%.

Based on the research conducted by Ji Zhang and his colleagues, it was expected that the resistance level against the tensile strength of the resin sample without nanoparticles, at a temperature of 80°C, would be 60 MPa with a strain of 3%.

Table No.1

Comparison of Tensile test Result

Nano Particle Weight percentage	Pure Resin Tensile Strenght (Mega Pascal)	Tensile Strength progress After Adding Nano Particles/Pure Resin Tensile Strength (Percentage)
0	62.36	۰
0.05	71.09	13.9
0.1	87.06	28.8
0.2	73.07	17.17

In the analysis of the Reactivitytests, it was determined that the sample with the highest heat peak of 1.0 had the highest level of network formation. This result was also obtained from the tensile tests of the samples, and factors a and b (which had the highest values in this sample in the responsiveness test) are again indicative of better completion of the polymerization process of the samples.

Table No.2

Comparison of Reactivity test results.

Nano Particle Weight percentage	Exothermic peak	A Factor	B Factor	Gel time
0 %	139.5	17:00	25:00	19:00
0.05 %	145.5	17:00	26:00	19:00
0.1 %	151.9	19:00	29:00	22:30
0.2 %	150.1	18:00	27:00	20:15

Using graphene oxide nanoparticles, a very high increase in its concentration prevents resin from curing. In such a way that at concentrations higher than 2.0%, curing does not occur.

A concentration of 1.0% of graphene oxide nanoparticles has the highest strength and improvement in mechanical resistance compared to other studied percentages in this research.

The peak exotherm in the reactivity test increases with an increase in the amount of nanoparticles up to 0.1%, which is due to a more complete polymerization reaction and better network formation.

Increasing the amount of nanoparticles (in this research up to 0.2%) leads to a decrease in network formation to the point that with an increase in the amount of nanoparticles up to 0.4%, the polymerization process does not even reach the gelation stage.

Suggestions

Based on the work done in this research, the following actions can be continued to achieve a more comprehensive goal:

For better results, it is recommended to first mix the nanoparticles and resin separately in a solvent using a mechanical mixer, and then perform ultrasonication by mixing both samples together.

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