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Mohanad H. Mohammed [1]*
Khaled W. Yahya [1]
Laith Y. Yaseen [1]
Mohammad M. Mahmood [2]
Abdulkhaliq A. Sulaiman [3]

[1] Nineveh Education Directorate, Ministry of Education, Mosul, 41200, IRAQ
[2] Department of New and Renewable Energy, College of Science, Mosul University, IRAQ
[3] Department of Physics, College of Science, Mosul University, Mosul, IRAQ
*Corresponding author Email: mohannad.hazim1983@gmail.com

Effect of Calcium Oxide Nanoparticles on Thermal and Mechanical Properties of Unsaturated Polyester Resin

In this research, calcium oxide (CaO) nanoparticles have been prepared using chemical bath deposition and characterized by the X-ray diffraction (XRD) and Raman spectroscopy, which show that the grains were within the nanoscale. The effect of calcium oxide (CaO) nanoparticles concentration on the mechanical and thermal properties of unsaturated polyester were investigated. The mechanical properties includes modulus of elasticity, compressive strength, impact resistance and hardness. The maximum values of modulus of elasticity, compressive strength, impact resistance and hardness were 834.79 MPa, 123.7, 44 kJ/m^2 and 122.9, respectively. The thermogravimetric analysis (TGA) curves show that the maximum onset and end decomposition temperature were observed at 8 g calcium oxide (CaO) nanoparticles. The thermal conductivity coefficient was also affected by calcium oxide (CaO) nanoparticles, where it decreased nanoparticles concentration.

Keywords: Composite materials; Calcium oxide nanoparticles; Unsaturated polyester; Hardness
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1. Introduction

The general definition of composite materials is that they are a mixture of two or more materials, each one possesses different physical and chemical properties, to prepare a new material with unique properties that exceed the properties of each of its constituent materials [1-3]. Composite materials are classified based on the base material type into metal-based, ceramic-based, or polymer-based composite materials [4,5]. Polymer-based composite materials are the most commonly composite materials because they have superior mechanical and physical properties [6,7]. They are also durable and resistant to internal and external stresses, and are more resistant to environmental conditions in terms of temperature and pressure than metals [8,9]. Composite materials consist of a combination of a base material, and reinforcing material (natural or synthetic) in the form of flakes, fibers, granules, or laminate [10-13]. Polymer-based composites are advanced materials used in a wide range of applications (biomedical, automotive, and construction materials) [14-18]. These composites are characterized by relatively high toughness, low density, and a strong glass-like structure with high toughness [19-21].

In this study, composite-based composite materials were manufactured, where unsaturated polycarbonate was used as the base material, and nano calcium oxide (CaO) was used as the reinforcing material. The effect of nano CaO concentration on the mechanical properties and thermal conductivity of composite materials was explored.

2. Materials and Methods

Unsaturated polyester resin (UPE) is a clear, viscous liquid at room temperature, with a specific gravity of 1.17 and a viscosity of 350-500 cps at 25°C. It solidifies upon the addition of ethyl methyl ketone peroxide, a colorless liquid (as an initiator for the polymerization process), and tin 2-ethyl hexanoate, a purple, oily liquid (as an initiator for dissolution). The hardener is added to the resin at a rate of 2.5% at laboratory temperature.

The reinforcing material is calcium oxide (CaO) nanoparticles, which is a white and non-toxic. Many techniques have been used to prepare nano CaO, but the best and easiest method is the chemical bath method, where calcium chloride ($CaCl_2$) is mixed with sodium hydroxide (NaOH) at weights calculated based on the molecular weight of each compound and the two compounds are dissolved in water. We dissolve 0.4 g of $CaCl_2$ and 0.5 g of NaOH in 200 ml of distilled water separately, then the two solutions are mixed to obtain calcium oxide as a precipitate, as in the following equation [22-26]:

$$CaCl_2 + 2NaOH \rightarrow Ca(OH)_2 + 2NaCl$$

The powder is then heated to obtain CaO nanoparticles.

Plastic molds were manufactured to prepare samples suitable for measuring mechanical properties.

The mold walls were coated with local grease to prevent the samples from sticking. Glass plates covered with greaseproof paper were placed under the molds to prevent the samples from sticking to the base, and the sample preparation process was completed by polishing them.

Hardener 2.5% was added to the unsaturated polyester material and mixed for four minutes to avoid the formation of air bubbles, which could led to sample failure. Different proportions of CaO nanoparticles powder (2%, 4%, 6%, and 8%) were mixed with unsaturated polyester for 1 min, the samples were left in the molds until they were set. After removing the samples from the mold, they were left for at least 30 hours to increase the bonding between the particles.

The samples were prepared with dimensions of 10×10×75cm according to American specifications (ASTM-D256-87) as shown in Fig. (1). The device used to measure the impact force is located in the College of Engineering, Mechanical Engineering Department.

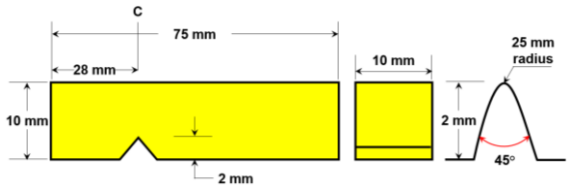

Fig. (1) V-notched IZOD specimen [27]

The hardness test is performed by placing the device perpendicular to the surface of the sample, and inserting the needle into the surface of the material (Fig. 2). The test was performed using a German Wolpert Shore-D device at the College of Engineering, Civil Engineering Department. In order to perform the compressive strength test, the samples were pressed into cylindrical shapes to be suitable for testing using a device located at the College of Civil Engineering, University of Mosul.

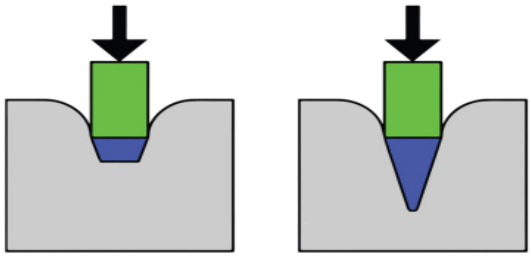

Fig. (2) Schematic explanation of hardness test

For elastic modulus testing, the samples were prepared as shown in Fig. (3). The device used is the Universal Test Machine. This test is used to determine the properties of a composite material under a two-way axial tensile load. The sample is securely clamped by the upper and lower jaws of the device, after which a tensile force is applied to the sample until it fractures.

For thermal conductivity measurements, the Lee disc device, shown in Fig. (4), was used in the College of Science, Physics Department. The samples prepared with 11.23cm diameter and 1 cm thickness, as shown in Fig. (8).

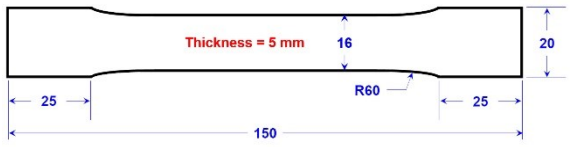

Fig. (3) A schematic drawing showing the dimensions and shape of the sample used to measure the modulus of elasticity [28]

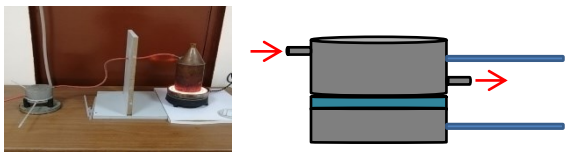

Fig. (4) Thermal conduction device

3. Results and Discussion

Calcium oxide (CaO) nanoparticles have been prepared using chemical bath deposition. The XRD spectrum show several peaks but the strongest peak was appeared at 2θ =18.99°, the structure was cubic with (110) orientation as indicated in Fig. (5) and table (1) [29]. The average grain size was about 22.4 nm.

Figure (6) shows Raman spectrum of prepared calcium oxide (CaO) nanoparticles. It shows several peaks at 331, 563, 731, 1000 and 1148 cm^{-1}. These peaks belong to calcium oxide (CaO) nanoparticles [30,31].

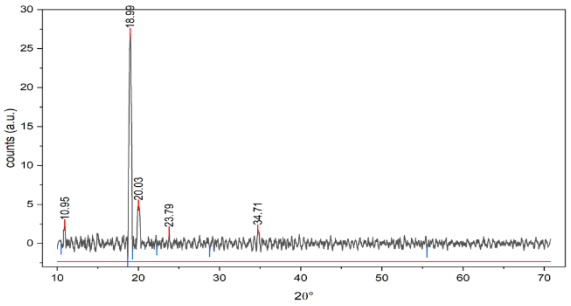

Fig. (5) XRD pattern of calcium oxide (CaO) nanoparticles

Table (1) Average grain size of calcium oxide (CaO) nanoparticles

2θ (deg)	FWHM (deg)	Crystallite size D (nm)	Average grain size (nm)
10.95	0.31030	25.7222	22.4244
18.99	0.36375	22.1456	
20.03	0.43785	18.4264	
23.79	0.31490	25.7840	
34.71	0.41528	20.0437	

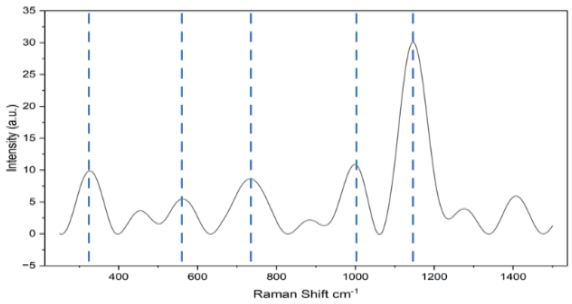

Fig. (6) Raman spectrum of calcium oxide nanoparticles

The elastic modulus of the unsaturated polyester was measured and compared with the elastic modulus of the polyester after it was reinforced with different weights of calcium oxide. We note that the lowest value of the elastic modulus was measured for the unsaturated polyester, and the elastic modulus increased with the increase in the weight of the added calcium oxide to its highest value of about 834.79 MPa at a weight of 8 g (see Fig. 7 and table 2).

The compressive strength of unsaturated polyester was measured before and after reinforcement with calcium oxide nanoparticles. We observed that the compressive strength increased with the increase in the weight of the calcium oxide nanoparticle reinforcement, as shown in Fig. (8) and table (3). The increase in compressive strength is due to the strong bond between the base material, the reinforcement, and the applied load, which in turn leads to an increase in the compressive strength.

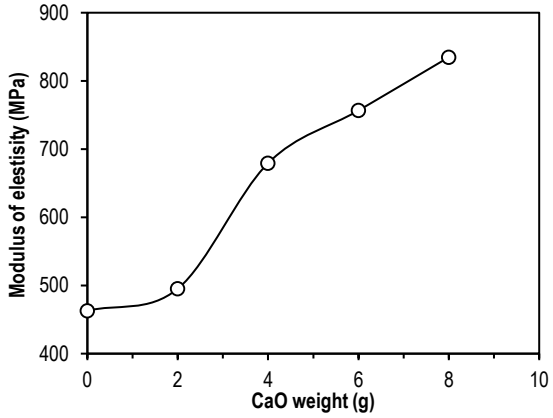

Fig. (7) Modulus of elasticity as a function of calcium oxide concentration

Table (2) Modulus of elasticity as a function of calcium oxide concentration

CaO nanoparticles Concentration (g)	Modulus of elasticity (MPa)
0	462.91
2	495.23
4	679.47
6	756.55
8	834.79

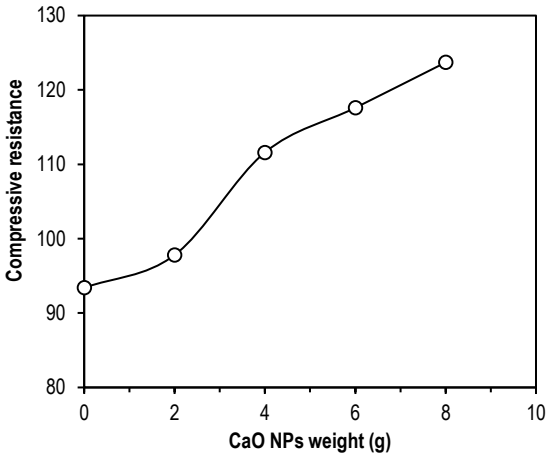

Fig. (8) Compressive strength as a function of calcium oxide (CaO) nanoparticles concentration

Table (3) compressive strength as a function of calcium oxide (CaO) concentration

CaO NPs Concentration (g)	Crystallite Size (nm)
0	93.4
2	97.8
4	111.6
6	117.6
8	123.7

The impact resistance of untreated polyester was measured before and after reinforcement. We found that the impact resistance increased with increasing weight of calcium oxide (CaO) nanoparticle reinforcement, with similar values. This is due to the large surface area of the nanoparticles, which allows them to withstand most of the applied stresses. These stresses are distributed over a larger area, reducing their impact on the composite material and, consequently, not affecting any cracks or weak spots in the sample, as shown in Fig. (9) and table (4).

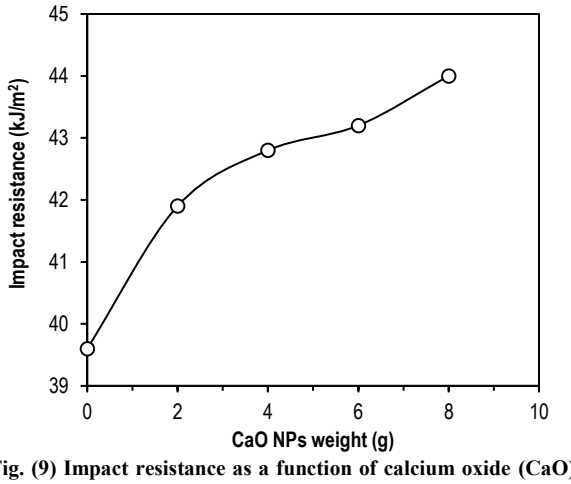

Fig. (9) Impact resistance as a function of calcium oxide (CaO) nanoparticles concentration

Table (4) impact resistance as a function of calcium oxide (CaO) nanoparticles concentration

CaO NPs Concentration (g)	Impact resistance (kJ/m²)
0	39.6
2	41.9
4	42.8
6	43.2
8	44

The hardness of the reinforcement samples was measured before and after reinforcement. We found that the hardness, like other properties, improved with increasing weight of calcium oxide (CaO) nanoparticles. This is due to a number of points, the most important of which is that the properties of the additives improve the properties of the base material. Since calcium oxide (CaO) nanoparticles are characterized by high hardness, they will consequently lead to improving this property of the samples, the strong bond between the unsaturated polyester and the reinforcement materials due to the small dimensions of the nanoparticles and the homogeneous distribution of the reinforcement materials, as shown in Fig. (10) and table (5).

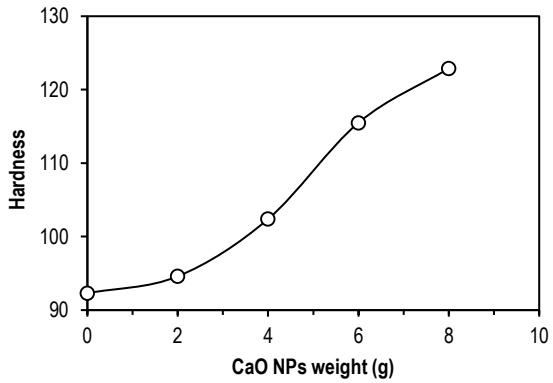

Fig. (10) Hardness as a function of calcium oxide (CaO) nanoparticles concentration

Table (5) hardness as a function of calcium oxide (CaO) nanoparticles concentration

CaO NPs Concentration (g)	Hardness
0	92.3
2	94.6
4	102.4
6	115.5
8	122.9

Figure (11) shows the thermogravimetric analysis (TGA) curves of pure unsaturated polyester and those reinforced with different weights of calcium oxide (CaO) nanoparticles. Slight differences were observed between the TGA curves of the pure polyester and those of polyester reinforced with low weights of reinforcement material, but the difference was clearer at high concentrations of calcium oxide (CaO) nanoparticles. The onset decomposition (IDT) and end decomposition (CDT) temperatures were determined by plotting the tangents to the TGA curves at the transition points with the straight part of the curve at the onset and end of the decomposition for the polymer composites. The results showed a clear change in the decomposition temperatures depending on the calcium oxide (CaO) nanoparticles concentration, as shown in table (6).

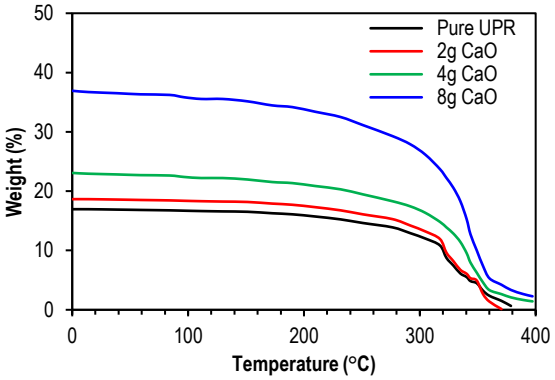

Fig. (11) Thermogravimetric analysis curve of unsaturated polyester as a function of calcium oxide (CaO) nanoparticles concentration

Table (6) Temperatures for the onset and end of untreated polyester resin with calcium oxide (CaO) nanoparticles concentration

CaO NPs concentration (g)	IDT	CDT
0	245	275
2	250	278
4	258	295
8	261	310

We note that the thermal conductivity decreased with the calcium oxide (CaO) nanoparticles concentration due to the nature of the reinforcement materials as they are heat insulating materials, in addition to effect of small particle size and reinforcement particles distribution within the composite. The reinforcement materials impede vibrations in the internal structure of the composite, and thus the conductivity coefficient values decrease, as shown in Fig. (12) and table (7).

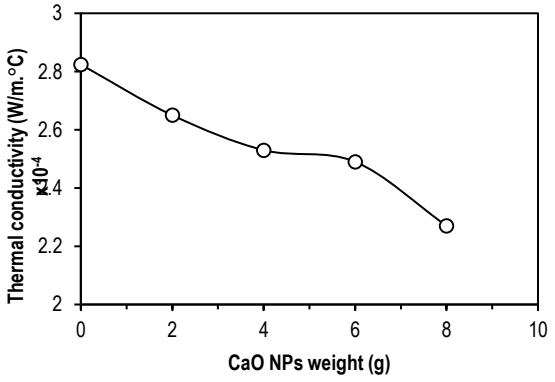

Fig. (12) Thermal conductivity coefficient of unsaturated polyester before and after reinforcement with different concentrations of calcium oxide (CaO) nanoparticles

Table (7) Thermal conductivity coefficient of unsaturated polyester before and after reinforcement with different concentrations of calcium oxide (CaO) nanoparticles

CaO NPs concentration (g)	Thermal conductivity coefficient
0	2.824×10^{-4}
2	2.65×10^{-4}
4	2.53×10^{-4}
6	2.49×10^{-4}
8	2.27×10^{-4}

4. Conclusions

Calcium oxide (CaO) nanoparticles have been prepared using chemical bath deposition. The grains were within the nanoscale with average grain size of about 22.42 nm and cubic crystalline structure with preferred orientation of (110). All mechanical properties of unsaturated polyester resin (UPR) were improved with increasing calcium oxide (CaO) nanoparticles concentration. The thermal properties of the pure and reinforced unsaturated polyester resin were also affected by adding calcium oxide (CaO) nanoparticles concentration. The maximum onset and end decomposition temperature were observed at 8 g of calcium oxide (CaO) nanoparticles. The thermal conductivity coefficient decreased with increasing calcium oxide (CaO) nanoparticles concentration.

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