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Mechanical Properties of Unsaturated Polyester Filled With Silica Fume, Glass Powder and Carbon Black

Abstract-In this study a polymer matrix composites (PMCs) have been prepared with the aid of ultrasonic wave dispersion method for mixing, using of unsaturated polyester resin with Silica Fume(SF), Glass Powder (GP) and Carbon Black (CB). Moulds were prepared by hand-made from silicon rubber according to the ASTM standard table (4). The fillers added separately with different ratios as (0%, 0.5%, 1%, 1.5 %, 2%, 2.5% and 3%). The results show increase the hardness and impact strength when added GP, SF, and CB to polymer matrix. Flexural strength and maximum shear stress decrease when added silica fume, but when added glass powder and carbon black led to increase flexural strength and maximum shear stress to certain percent at (2%, 1.5% for GP and CB respectively) then dropped when increase weight fraction of GP, CB. Compressive strength decrease when added glass powder and carbon black, while it is increase when added silica fume to polymer matrix. Wear rate decrease when increase weight fraction of carbon black but it increases when added GP and SF.

Keywords- mechanical properties, unsaturated polyester resin.

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1. Introduction

Composites are characterized as materials which incorporate at least two or more physical and chemical various phases, the distinct interface separated between them. The various systems combine with each other to form a system possesses the properties of structural and functional more than constituent alone. Composites, are wonderful materials have become a basic part of today's materials because of the advantages possessed by such as low weight, high fatigue strength, corrosion resistance, assembly faster, which is used widely as materials in the manufacture of the shuttle space structures, packaging to medical equipment and construction [1].

Flores et al. studied the influence of filler structure onto micro-hardness (H) for low density Polyethylene-carbon black and polycarbonate - carbon black composites. They were used two kind from microadditives at various average particles sizes. It was found that the morphology of the polymer matrix affect on the hardness of composites which depend on the composites and the volume concentration for filler. The micro-hardness for polycarbonate-carbon black composites shown a step like behavior regarding to content of carbon black, whilst the H values for low density polyethylene-carbon black composite linear increasing with increase volume concentricity for filler. Their Results shown the smaller particle size of carbon

black enhances the micro hardness of the composites [2].

Hassan et al. developed polyester/eggshell particulate composites, he was utilized carbonized and uncarbonized eggshell particles as reinforcing into polyester matrix. Eggshell particles at (10%, 20%, 30%, 40% and 50%) of weight were add to polyester as reinforcing material. The microstructural analysis for the particulate composites (polyester/eggshell) was carried out utilization a scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS). Results shown the hardness and density for the particulate composite increased with increasing the addition of eggshell. The bending strength and tensile for the composite increased with increasing for weight of eggshell particles into polyester matrix, from the SEM detected better strengthening effect of carbonized eggshell due to better interfacial bond between polyester matrix and carbonized particles [3].

Alkhafaji studied the mechanical and electrical behavior for composites from polymer matrix and their hybrids, particles of carbon black used as reinforcing material at constant volume fraction (10) % and boron particles at various volume fractions (0%, 2%, 4%, 6%) which are bounded with unsaturated polyester resin. Results showed increasing in hardness values with volume fractions of boron powder. It is observed the values of hardness are increased with increasing volume fraction of reinforced boron

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particles to matrix due to the boron particles have excellent hardness compared with black carbon particles, in hybrid composite material: polyester resin reinforced with hybrid particles of (black carbon-boron), the hardness increase with contain of boron [4].

Saleh et al. studied mechanical properties of epoxy filled with fly ash and silica fume added each filler separately with various ratios as (10%, 20%, 30%, 40%, 50%), their results showed that the increase of additives ratios of silica fume and fly ash caused increasing the compression strength and tensile strength, the increase of additives ratio of silica fume causes decreasing in bending strength [5].

Musa studied the influence of adding glass powder of grain size (35µm) with various volume fraction (10%, 20%) to the blend of unsaturated polyester and polyurethane. Her results have shown that the addition of particles to the polymer blends lead largely in mechanical properties it has shown that the values of young modulus, impact strength and hardness (shore D) were increase with increasing of volume fraction of glass powder. Fracture surface of the samples were examined using optical microscope with magnification (40 X) and the results showed that the nature of fracture is seems brittle fracture for all samples [6]. This work aimed to evaluate the mechanical properties (hardness, bending, impact strength, compression strength, and wear) for unsaturated polyester as a matrix with silica fume, glass powder and carbon black as reinforcement composite systems.

Nomenclature

A	Cross-section Area(m ²)	R	Radius from the center of sample to the disk center (cm)
b	Width of sample (m)	T	Sliding time (s)
H	Thickness of sample (m)	UC	Fracture energy (J)
FS	Flexural Strength (MPa)	W.R	Wear Rate (g/cm)
IS	Impact Strength (KJ/m ²)	Wt%	Weight Fraction %
L	Length of specimen(m)	τ	Maximum Shear Stress (MPa)
m ₁	Weight of sample before test (g)	σ _c	Compressive Strength (MPa)
m ₂	Weight of sample after test (g)	CB	Carbon Black
Δm	Mass loss (g) = m ₁ - m ₂	GP	Glass Powder
N	Number of disk rotating (cycle/min.)	PMCs	Polymer Matrix Composites
P	Load of Fracture (N)	SF	Silica Fume
p _c	Compressive Load(N)	UP	Unsaturated polyester

2. Experimental Work

I. Materials

1. Matrix Material

Unsaturated polyester resin is a kind of thermosetting polymer; it was utilized as a matrix material. Unsaturated polyester of a trademark (Siropol8340) is a viscous liquid resin that converts to a solid state using curing catalyst (as preparation of PMC), accelerator cobalt naphthenate was added into the resin matrix at percentage 0.5g for 100 g of resin according to the supplying company. Curing agent is methyl ethyl ketone peroxide (MEKP) at a concentration of 2%wt. of the matrix. All these materials were supplied by Saudi Industrial Resins Ltd (Saudi Arabia Company). This resin system was selected because availability and low cost. Table 1 shows the unsaturated polyester characteristics according to Manufacturing Company.

Table 1: Unsaturated polyester properties according to product company

Density (g/cm ³)	1- 3 at [25C°]
Tensile strength (MPa)	41.4 – 89.7
Percent Elongation %	< 2.6
Fracture Toughness (MPa-m ^{0.5})	0.6
Thermal Conductivity(W/m. K°)	0.17
Modulus of elasticity (GPa)	2.06 – 4.41
Particle size	45 µm

2. Reinforcing Materials

a. Silica fume

Silica fume (SF) is a by-product of melting process in ferrosilicon and silicon industry. The decrease for high pureness quartz to silicon at temperature up to 2000 C° output SiO₂ vapour, that oxidizes and condense at lower temperature zone to teeny particles consist of noncrystalline silica. Table 2 shows the typical characteristics of silica that used in this research.

Table 2: The typical characteristics of silica fume that used in this research

Property	Value
State	Amorphous sub-micron powder
Colour	Grey to medium grey powder
Specific Gravity	2.10 to 2.40
Bulk Density	500 to 700 kg/m ³
Silicon Dioxide (SiO ₂)	Minimum 85%
Moisture content(H ₂ O)	Maximum 3%
Specific Surface Area	Minimum 15 m ² /g

b. Carbon Black

Carbon black is a material produced by an insufficient combustion from heavy products of petroleum. Carbon black is a take shape of amorphous carbon that has a high surface to area ratio. Traditionally, carbon black is utilized as a reinforcement agent in tires. Today, due to the unique properties owned by carbon black, has expanding its uses to include pigment, stabilizing ultraviolet light (UV), and conductive agent in a variety of every day and specialty products of high performance which include: tires, products of industrial rubber, electrical discharge compounds, high performance coating, ink and toners printing [7].

c. Glass Powder

Glass is a solid material generally composed of noncrystalline silica, calcium oxide, sodium oxide and another components. The chemical composition rely on the raw materials utilized and vary slightly from each kind of glass. Glasses generally refer to hard, brittle, transparent material; have low ductility, low thermal expansion and low thermal conductivity so it has low resistance to thermal shock. Glasses have a resistant to many solvents, acids and other chemicals [6]. Glass powder used in this research was prepared from the cups transparent glass; it was crushed to small pieces and then milled to micro-scale (75µm).

3. Preparation Technique

I. Mould Preparation

The moulds of specimens used in the compression, bending, and hardness, impact and wear test are fabricated from silicone rubber, The shape and dimension of all moulds are fabricated according to the standard dimensions (ASTM) for each test. Samples dimensions shown in Table 4.

II. Preparation of polymer matrix composites

Polymer matrix composites (PMCs) were prepared from additives (silica fume, glass powder and carbon black) filled unsaturated polyester matrix at different weight fraction (0, 0.5, 1, 1.5, 2, 2.5, 3) %. In the present work, the ultrasound technique is used for manufacturing of PMCs due to its efficiency in breaking the agglomerating of microparticles and making them homogeneously dispersed in the polymer matrix, as well as, to ease

of cleaning tools. The steps of samples manufacture can be describe as following:

1- Weighing of silica fume, glass powder, and carbon black according to the required weight fraction (0, 0.5, 1, 1.5, 2, 2.5 and 3) Wt.% of unsaturated polyester resin.

2- Silica fume, glass powder, and carbon black mixed by hand with UP resin around (20) minutes at room temperature constantly and slow to obtain a homogeneous mixture and to avoid bubbling during mixing.

3- Putting the mixture in an ultrasonic wave bath machine [model (BK-9050) (50 W for 30 min.), (power source : 220 V AC\ 50 Hz)]as show in Figure 1, for 30 minutes to disperse the particles in polymer matrix homogeneously and to avoid the generation of heat through mixing that is influence onto properties of UP.

4- Adding the hardener (methyl ethyl ketone peroxide MEKP) to mixture at 2% Wt.of unsaturated polyester resin, then the mixture is poured into the mold until it is filled on a regular basis. Left the mixture into a mold for (24 hours) at room temperature for solidification.

5- Put the cast in drying oven (Post Curing) at 50 ° C for 2 hours, this process is very important to complete the polymerization, remove residual stresses and obtained best coherency. Working in a laboratory in the Department of Materials Engineering, Al-Mustansiriyah University.

4. Mechanical Tests

I. Impact test

The charpy impact test on unmatched samples was studied by use pendulum impact testing device (HSM41 Pendulum Impact Tester), it was made in German. The test is done according to ISO-179 standard with dimensions of specimen: length (55mm), width (10mm), and height (5mm).Dimensions of specimen can be calculate from equation: (A=b*h) where A is the cross - sectional area of the specimen, b, h are the width and thickness of the sample respectively. Hammers with (5 Jules) fracture energy are used, weight of hammer used in this research is (2.5) Kg.

$$IS = \frac{UC}{A} \tag{1}$$

Table 3: Chemical composition for glass powder

components	SiO ₂	Na ₂ O	K ₂ O	MgO	CaO	Al ₂ O ₃	Fe ₂ O ₃
Weight %	52.5	9.10	7.20	1.50	0.56	0.75	0.18

Table 4: Dimensions and standard specifications for the testing specimens

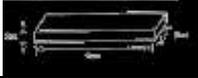
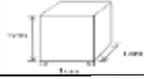
Test	Sample dimensions	Standard Specifications
Impact		ISO-179
Bending & Flexural Strength		ASTM-D790
Compression		ASTM-D695
Wear		ASTM- G99
Hardness		ASTM -D2240



Figure 1: Ultrasonic wave bath machine



Figure 2: Impact samples

II. Hardness test

This test was carried out utilize a Digital Micro Shore D (Durometer) (QUALITEST HPE) device according to (ASTM D2240), it is manufactured in USA. Seven measurements of hardness were made at different positions on the specimens to determine the average value.



Figure 3: Hardness samples

III. Bending Test

This test is called three points test, the bending tests were performed according to ASTM-D790 standard with dimensions of sample: length (100mm), width: (10 mm) and height: (5mm). The test was carried out using the universal mechanical test machine (Model RH1 5DZ, Tiniusolsen Ltd), it was made in England. The main purpose of this test is to calculate the maximum flexural strength and maximum shear stress by equations (2) and (3) respectively:

$$FS = \frac{3PL}{2bh^2} \tag{2}$$

$$\tau = \frac{3P}{4bh} \tag{3}$$

IV. Compression Test

Specimens were presented according to standard (ASTM D 695) at room temperature with a speed rate of (1mm/min), as shown in table (3). The test was done using the universal mechanical test machine (Model RH1 5DZ, Tiniusolsen Ltd), it is made in England. The stress can be calculated by the following equation:

$$\sigma_c = \frac{Pc}{A} \dots\dots\dots(4)$$



Figure 4: Bending samples



Figure 5: Compression samples



Figure 6: Wear samples

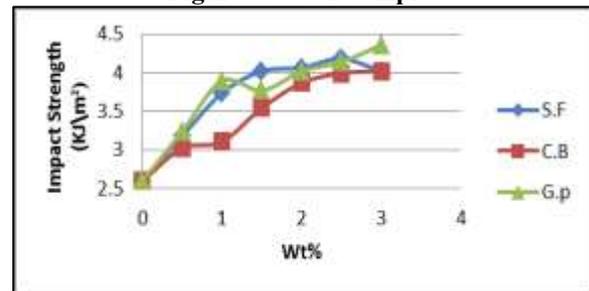


Figure 7: Effect of glass powder, silica fume and carbon black on impact strength of unsaturated polyester resin

V. Wear Test

In this test, specimens were prepared according to ASTM-G99. A pin on a disk was used to examine the wear test. An abrasive disk (tool steel) with hardness (HV=277) and speed (950 rpm). The load (5 N) was applied for different times (2, 4, 6 min). Radius from a center of specimen to the center of disk is (6cm) for each specimen. The equation (5) could be used to calculate the wear rate.

$$W.R = \frac{\Delta m}{SD} \quad (5)$$

$$SD = 2\pi rnt$$

5. Results and Discussions

I. Impact strength

Figure 7 indicating the relation between the impact strength and the weight fraction for GP, SF, and CB that are added to the UP matrix. Results have detected that the maximum amount of impact strength (4.357kJ/m² at 3%), (4.01762.598 kJ/m² at 3%) and (4.181kJ/m² at 2.5%) for GP, CB and SF respectively, compared to the impact strength of the neat (2.598kJ/m²). The increase in the concentration of the filler increase the ability of matrix to soak up energy and that way increases the toughness, so that impact strength is increases. It is observed that the filler with smaller particle size show a higher increase of impact strength. The dropping in the impact strength value may be attributed to low the adhesion between the particulate filler with the matrix or the presence the tiny voids within the sample, which led to a decline in values of impact strength.

II. Hardness Test

Hardness is a measure of the resistance to penetrate the surface, as they are a function of stress required to produce some certain types of deformation of the surface [8].

Figure 8 indicating the relation between the hardness shore D and a weight fraction for GP, SF and CB tha are added to UP matrix.

Results show a higher value for hardness shore D is (87.93 at 3%), (84.16at 0.5%) and (84.21at 3%) for GP, SF, CB respectively, compared to the hardness shore D of the neat (65). The increase in hardness values with increase weight fraction of glass powder and carbon black agree with results of Musa [6] and Flores [9].

Hardness values increased for all samples which strengthen by glass powder, silica fume, carbon black due to increased crosslinking and stacking of the unsaturated polyester matrix (which reduces the. movement. of polymer molecules), which led to increased resistance to scratching material and cutting, thus increase hardness values.

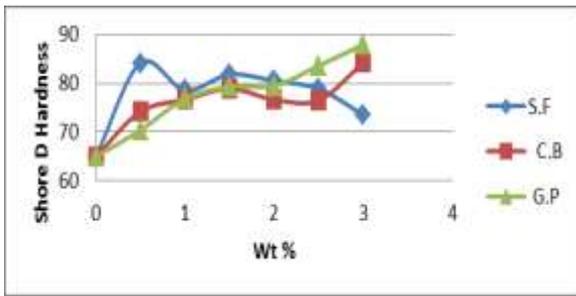


Figure 8: Effect of glass powder, silica fume and carbon black on hardness shore D of unsaturated polyester resin

III. Bending test

Bending describe the behavior of a structural element subjected to an outer force applied vertical to the axis of the element [10]. Figure 9 shows the relation between the bending (flexural) strength and the weight fraction of GP, SF and CB that were added to UP resin. From results show decrease in flexural strength when adding SF as filler, while flexural strength increase to (665.61Mpa at 2%) and (383.9Mpa at 1.5%) when added GP, CB respectively, and then dropped when increase in weight fraction of GP, CB. The increase in flexural strength may be attributed to a small amount of particles dispersed homogenously in unsaturated polyester, that leads to a strong interface between particles surface and unsaturated polyester because of its own higher surface area, and thus got improved in the flexural strength.

Figure 10 shows the relation between the shear stress and weight fraction of the GP, SF and CB, from the Figure 10 showed particle-reinforced composites, most of these composite materials, the particulate phase is harder and stiffer compared to the matrix. These reinforcement particles tend to restrict the movement of the matrix phase in the closeness of each particle. The principle of the matrix transfers some of the applied stress to the particle. The degree of reinforcement or improvement of mechanical behavior depends on strong bonding at the matrix-particle interface [11].

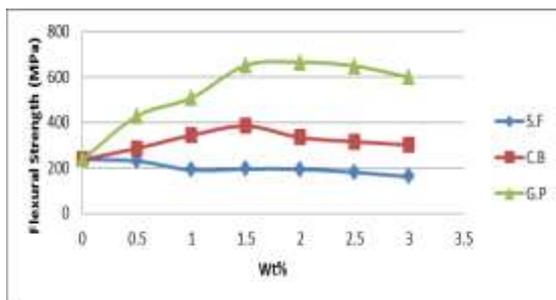


Figure 9: Effect of glass powder, silica fume and carbon black on flexural strength of unsaturated polyester resin

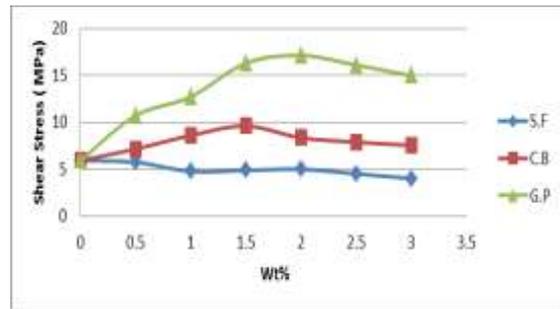


Figure 10: Effect of glass powder, silica fume and carbon black on shear stress of unsaturated polyester resin

IV. Compression Test

This test includes an axial compression force applied on the compression standard specimen with a square cross- section. Figure 11 shows the effect of GP, SF, and CB at various loading level on compressive stress of unsaturated polyester matrix. The results show decreasing in compressive strength when added GP,CB to unsaturated polyester matrix this may be due to the particles (reinforcing) capability, which do not have high compressive strength, while compressive strength is increase to (159.04 Mpa at 1.5%) when added SF as compare to the compressive strength of pure unsaturated polyester resin (133.03 Mpa). The increase in compressive strength attributed to SF has high compressive strength also a good interface between the filler and matrix. The compression strength for composite gradually decreases when the filler beyond (1.5 % wt.). Its indicates that the lower degree of particles-polymer interaction occur at higher filler contents because of the agglomerated of SF when content of filler increased.

V. Wear Test

One of the most important characteristics that determine the behavior of polymer composites is due to erosion because in the parts of the applications of polymer composite often operating in the industry [12].

Figures 12, 13 and 14 show the effect of GP, SF, and CB respectively at different loading level on the wear rate of unsaturated polyester matrix. Wear test has be done at different times (2, 4, 6 min.), constant speed (950 rpm) and load (5 N). The results show increase in wear rate with increase weight fraction and time when adding GP and SF to unsaturated polyester matrix as show in figures (12),(13) respectively, but remained lower than the wear rate of unsaturated polyester only. When added CB to unsaturated polyester matrix led to decrease in wear rate with increase the loading level and time. It means the

particles are very effective in improving the wear performance of UP. The improvement in abrasive wear may be attributed to the improved dispersion of the particles.

Properties of wear can be changed substantially by the change in the microstructure, mechanical properties of the reinforcing phase, the weight fraction, and the nature of the interface between reinforcement and matrix [9].

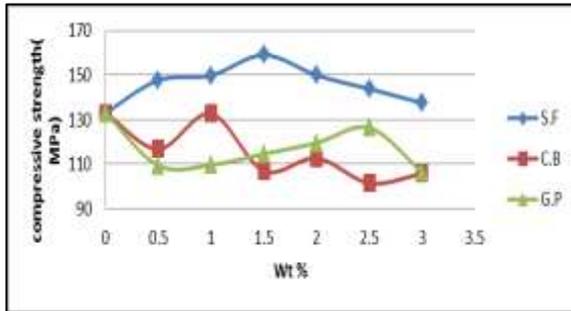


Figure 11: Effect of glass powder, silica fume and carbon black on compressive strength of unsaturated polyester resin

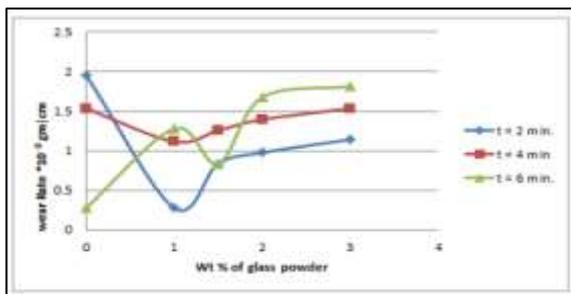


Figure 12: Effect of glass powder on wear rate of unsaturated polyester resin

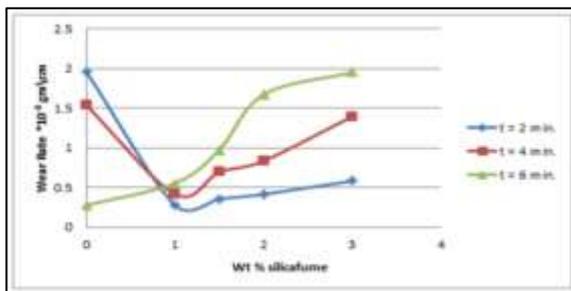


Figure 13: Effect of silica fume on wear rate of unsaturated polyester resin

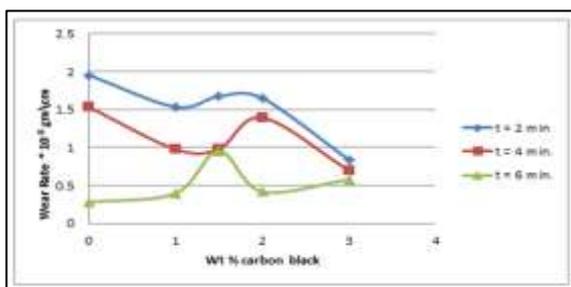


Figure 14: Carbon black effect on the wear property of unsaturated polyester

6. Conclusions

1. Hardness shore D increase with increase weight fraction of fillers added to unsaturated polyester matrix. The percentage of increase in the hardness of unsaturated polyester are (29.47%, 29.55%, 35.28%) for SF, CB, and GP respectively.
2. Impact strength increase with increase loading level of particles. The percentage of increase impact strength are (60.39%, 54.64%, 67.71%) for SF, CB, and GP respectively.
3. Flexural strength and maximum shear stress decrease when added silica fume, but when added glass powder and carbon black led to increase flexural strength and maximum shear stress to certain percent (at 2%, 1.5% for GP and CB respectively) then dropped when increase weight fraction of GP, CB.
4. Compressive strength decrease with increase weight fraction of glass powder and carbon black the percentage decreases (5.03%, 0.248%) for GP, CB respectively , while it is increase to percentage (19.55%) when added silica fume to polymer matrix.
5. Increasing the wear rate with increase weight fraction and time when added glass powder and silica fume to the polymer matrix, but wear rate decreases when increase weight fraction of carbon black .

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