International Journal of Engineering Science Invention (IJESI)
ISSN (Online): 2319 – 6734, ISSN (Print): 2319 – 6726
www.ijesi.org ||Volume 7 Issue 5 Ver VII || May 2018 || PP 56-63

Effect of Al_2O_3 – 40 wt. % TiO_2 ceramic particle size and shape on flexural properties of glass fibre reinforced epoxy composites

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Abstract: *The objective of this study is to investigate the flexural properties of glass fibre reinforced epoxy composite filled with different form (fused and crushed, agglomerated), size and proportions of Al_2O_3 – 40 wt. % TiO_2 particles. The composite plates are filled with 0% (unfilled), 5%, 10% and 15% particle weight fractions (based on the weight of resin). Composites are fabricated in conventional hand lay-up technique. Tensile and flexural properties of specimens were determined as per ASTM standards. The results indicate that the tensile and flexural properties of composites are significantly influenced by particle weight fractions, different particle sizes and forms. The results showed that while tensile strength and flexural strength of the composites decreased with increasing Al_2O_3 – 40 wt. % TiO_2 particles content, tensile modulus increased with the Al2O3 particles content. Compared with the flexural properties of the unfilled glass fibre reinforced epoxy composite, with the addition of 5 wt.% of Al_2O_3 – 40 wt. % TiO_2 particle in the matrix, flexural modulus were increased by 7%.*
Keywords**:** *Composite materials, Epoxy, Flexural properties, Glass fibre, Particle-reinforcement.*

Date of Submission: 20-05-2018 Date of acceptance: 02-06-2018

I. Introduction

Composite materials have extensively been used increasingly in modern engineering applications such as aerospace industries, automobiles, marine, and defense industries due to their good corrosion resistance, lightweight, and better mechanical characteristics than metals [1-3]. Epoxy resin as matrix is widely used in the production of glass fiber composites. However, the application of epoxy resin-matrix composites in some industries is usually limited owing to the relatively poor mechanical and wear-resistance properties. In order to enhance the strength, wear resistance and thermal stabilities, many studies have been carried out. One of these is modification of matrix. Many hard particulates made of ceramic particles have been tried as the fillers to modify the epoxy resin-matrix composites for that purpose by several workers[4-8]. Their results show that the addition of various ceramic particles into epoxy matrix enhances the fracture toughness, impact resistance and electrical or heat transfer properties, resin stiffness, wear resistance properties of the composites. In general, the mechanical properties of particulate filled polymer composites depend strongly on size, shape, particle/matrix interfacial adhesion and distribution of filler particles in the polymer matrix [9-10].

Although there has been considerable research devoted to the physical, thermal, mechanical properties and wear characteristics of unfilled glass fibre reinforced epoxy resin composites and ceramic or metal particles filled pure epoxy resin composites [4-20], there are not experimental data about the effect of ceramic particle size and shape on the flexural properties of glass fibre reinforced epoxy composite filled with different form, size and proportions of Al_2O_3 – 40 wt. % TiO_2 particles.

Therefore, in the present paper, an experimental study has been carried out to investigate the effect of ceramic fillers on flexural behavior of glass fibre reinforced epoxy composite filled with different form, size and proportions of Al_2O_3 – 40 wt. % TiO_2 particles. As a comparison, the mechanical properties of unfilled glass fibre reinforced epoxy composite were also evaluated under identical test conditions.

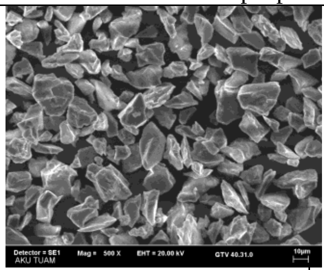
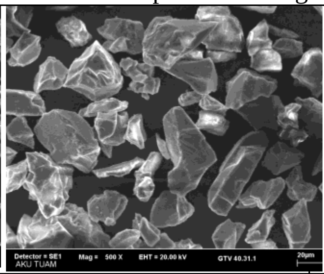
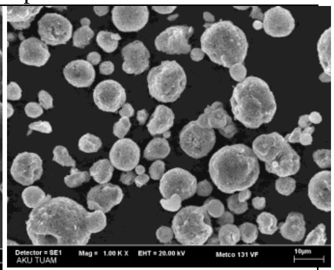
II. Ii. Materials And Experimental Procedures

2.1. Materials and Fabrication Laminates

Composites were fabricated in conventional hand lay-up technique. A commercially available plain-weave woven glass fabric with areal weight of the fabric is 270 g/m2 was used as reinforcement material. The type of epoxy resin used in the matrix material was Epikote Resin 828 and the hardener is Epikure Curing Agent 875. Epoxy resin and hardener are mixed in a ratio of 100:80 by weight as recommended by the supplier. The fillers are mixed with known amount of epoxy resin. As filler, three Al_2O_3 – 40 wt. % TiO_2 ceramic particles with different form, size and proportions were used to modify the epoxy matrix.

In order to investigate the flexural properties of glass fibre reinforced epoxy composite filled with different form, size and proportions of Al_2O_3 – 40 wt. % TiO_2 particles, the specimens were divided into four groups, namely Unfilled, Group A, B, and C respectively. The some properties and scanning electron micrograph of the used powders were given Table 1. A weighed amount of the Al_2O_3 – 40 wt. % TiO_2 particles was prepared based on the weight fraction of the particles to the total weight of the epoxy and Al_2O_3 – 40 wt. % TiO_2 particles mixture. The weight fractions of the filler in the matrix were 0%, 5%, 10% and 15%.

Table 1. Some properties of ceramic powders and groups.

SEM Micrograph of Ceramic Powders			
Sample Code	Group A	Group B	Group C
Ceramic Powder Content	Al_2O_3 – 40 wt. % TiO_2	Al_2O_3 – 40 wt. % TiO_2	Al_2O_3 – 40 wt. % TiO_2
Ceramic Powder Size	-25 + 5 µm	-45 + 20 µm	-45 + 5 µm
Ceramic Powder Type	Fused and Crushed	Fused and Crushed	Agglomerated

For the preparation of ceramic particles filled composites, first, epoxy resin was preheated to enable a better wetting of the particles. The epoxy resin was mixed with a hardener in a ratio of 100:80 by weight. After incorporating the fillers into the matrix material, the mixtures were carefully mixed by mechanical stirring. In order to obtain filled composite laminates, the glass fibre woven fabrics were put into a mould in the same directions and the mixture (consisting of epoxy, the Al_2O_3 – 40 wt. % TiO_2 particles and the hardener) impregnated. After the impregnation, the complete assembly was placed in a hydraulic hot press machine with a pressure of 15 MPa at a temperature of 120 °C applied during 3.5 h. At the end of the process, the complete set-up was cooled slowly to room temperature. Composite laminate thickness was approximately 2.5 mm. The unfilled glass fibre reinforced epoxy composite was fabricated in the same manner except that no Al_2O_3 – 40 wt. % TiO_2 particles fillers.

2.2. Density and void content measurement

The actual density of the composite is determined experimentally by simple water immersion technique as per ASTM D 792 standard [21]. Four samples of same composite are tested and average value is presented. The weight fraction of contents (fiber, resin and filler) has been determined as per ASTM D 2584 standard [22]. This test method can be used to obtain the ignition loss of a cured reinforced resin sample.

The void content of composite sample has been determined as per ASTM D-2734-70 standard [23]. The volume fraction of voids (V) in the composites was calculated using the following equation (1):

$$V = 100 - (Md \times ((\frac{r}{dr}) + (\frac{g}{dg}) + (\frac{t}{dt}))) \tag{1}$$

where:
V = void content, volume %,
Md = measured density,
r = resin, weight %,
g = glass, weight %,
t= filler, weight %,
dr = density of resin,
dg = density of glass and
dt = density of filler.

2.3. Mechanical testing

The tensile tests were conducted according to the ASTM D3039-76 standard [24]. The test specimens were cut with a circular diamond blade saw into the rectangle blank. In order to avoid catastrophic influence of surface flaws, the specimens edges were carefully finished using emery papers. Aluminum end tabs were bonded on the specimens for proper gripping and to ensure failure in the gauge section. The tensile specimen is placed in the testing machine, taking care to align to longitudinal axis of the specimen. Tests are conducted for the samples at normal room temperature. The specimens were loaded in tension at a constant stroke rate of 2 mm/min. One group of unfilled samples was also tested for comparison purpose. For each composition, five identical samples were tested and average results reported.

In order to determine the flexural properties of the composites three point-bending test was carried out according to ASTM D 790 standard [25]. All tests were carried out in a universal testing machine while recording load and deformation data on a computer, at room temperature. In each case five samples were used and the average properties were taken. Cross-head speed was 0.5 mm/min. One group of unfilled samples was also tested for comparison purpose. For each composition, five identical samples were tested and average results reported:

The flexural stress in a three-point bending test was found out by using the following equation (2):

$$\sigma_{f_s} = \frac{3.F_f.L}{2.b.t^2} \tag{2}$$

where F_f is the maximum load (N) at failure, L is the distance between the supports (mm), b and t are the width and thickness of the specimen (mm), respectively.

Flexural modulus was calculated using the following equation (3):

$$E = \frac{(m.L^3)}{(4.b.t^3)} \tag{3}$$

where 'm' is the slope of the linear portion of load–deflection plot, b and t are the width and thickness of the specimen (mm).

III. Results And Discussion

The variations of physical and mechanical properties of the glass fibre reinforced epoxy composites with Al_2O_3 – wt. 40 TiO_2 particle content were given in Table 2. Its corresponding data were plotted in Fig. 1-5.

Table 2. Some of the properties of the manufactured composite materials.

Group code	Filler ratio (%)	Thickness (mm)	Measured density (gr/cm³)	Void (%)	Fiber weight ratio (%)	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)	Flexural deflection (mm)
Unfilled	0	2,14	1,906	1,33	74	405	10,76	641	38,06	13,6
<u>Grup A</u> Al_2O_3 – 40 wt. % TiO_2 (-25+5 µm) (Fused and Crushed)	5	2,21	1,927	2,61	72	350	10,82	446	36,44	8,50
	10	2,23	1,954	2,74	71	325	10,67	457	34,06	8,30
	15	2,31	1,981	3,60	70	275	9,94	373	33,76	8,00
<u>Grup B</u> Al_2O_3 – 40 wt. % TiO_2 (-45+20 µm) (Fused and Crushed)	5	2,12	1,932	2,50	72	330	10,89	426	31,20	8,30
	10	2,42	1,959	2,83	71	308	11,07	419	33,61	7,60
	15	2,48	1,982	3,33	67	328	10,65	401	31,35	6,90
<u>Grup C</u> Al_2O_3 – 40 wt. % TiO_2 (-45+5µm) Agglomerated)	5	2,12	1,917	2,40	71	370	10,88	492	40,57	9,10
	10	2,27	1,924	3,45	69	315	10,67	424	37,24	8,20
	15	2,35	1,937	3,95	68	255	9,47	407	36,59	8,10

The properties of the produced composite materials are evaluated; it has been found that as the amount of ceramic powder added to the epoxy resin increases, the thicknesses, density and porosity values of the composite materials produced increase. It has been found that the produced composite materials have a fiber weight ratio of between 68 and 74 percent by weight, and the highest fiber weight ratio is found in the samples of unfilled glass fiber reinforced composite materials. It was found that the lowest porosity (1.33%) in the composite materials occurred in the samples of unfilled glass fiber reinforced composite materials, and the highest porosity (3.95%) was found in the samples with 15wt.% ceramic powder granules in Group C. The high amount of porosity in composite materials generally negatively affects the mechanical properties of the material. The variation of tensile strength and tensile modulus of the glass fiber reinforced epoxy resin composites with weight fraction of Al_2O_3 – 40 wt. % TiO_2 particle content are shown Fig.1-2. According to the results of the tensile tests of the samples, it was determined that the samples with no added ceramic powder had the highest tensile strength and the tensile strength of the materials decreased as the ceramic powder additive ratio in the produced composite materials increased. It was determined that only the tensile strength of the 15wt.% ceramic powder reinforced composite material in Group C was somewhat higher than the tensile strength of the 10wt.% ceramic powder reinforced sample. This may therefore be related to a stronger bond between the added ceramic powder particles and the resin matrix material. The highest tensile strength of ceramic powder filled composite materials with 5% ceramic powder adhering samples was found in the samples in Group C. In addition, when the tensile strengths of the samples in Group A and Group B with the same powder geometry are compared, the tensile strength of Group A samples with lower powder particle size is higher.

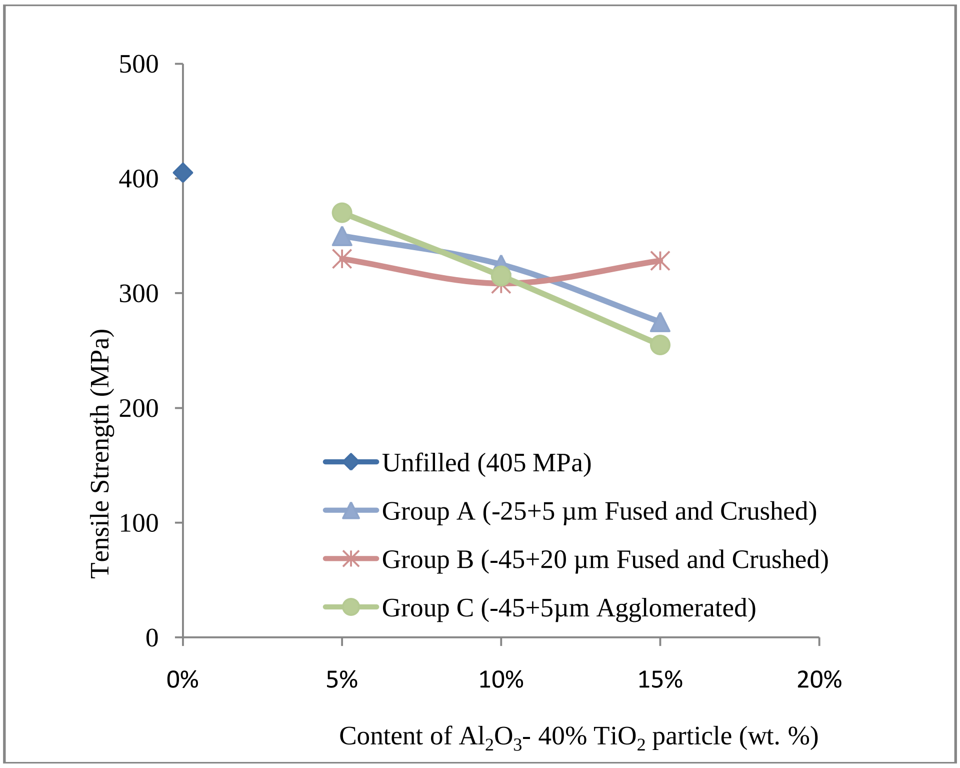


Figure 1. The variation of tensile strengths of the composites with weight fraction of particle content.

According to the result of the tensile tests, the values of the tensile modulus occurring in all 5wt.% of the ceramic powder filled samples are higher than those of the tensile modulus of the composite material. Generally, as the ceramic powder filler ratio in the resin matrix material increases, the tensile modulus values of the materials decrease. However, the highest tensile modulus in Group B materials was found to be in samples with 10wt.% ceramic powder filled. When the values of the tensile modulus of the samples in Group A and Group B with the same powder type were compared, the tensile modulus of Group B samples with larger powder particle size was higher.

Ceramic particles have a dramatic effect on the flexural strength and flexural modulus of the glass fibre reinforced epoxy composites. The variation of tensile strengths of the glass fiber reinforced epoxy resin composites with weight fraction of Al_2O_3 – 40 wt. % TiO_2 particle content is shown Fig.3-5. It is clearly seen that filled particle type, weight fractions and size in the composite materials appear to influence flexural properties of composites. According to the flexural test results of the composite materials; the highest flexural strength value of unfilled glass fibre reinforced epoxy composite was found in the samples.

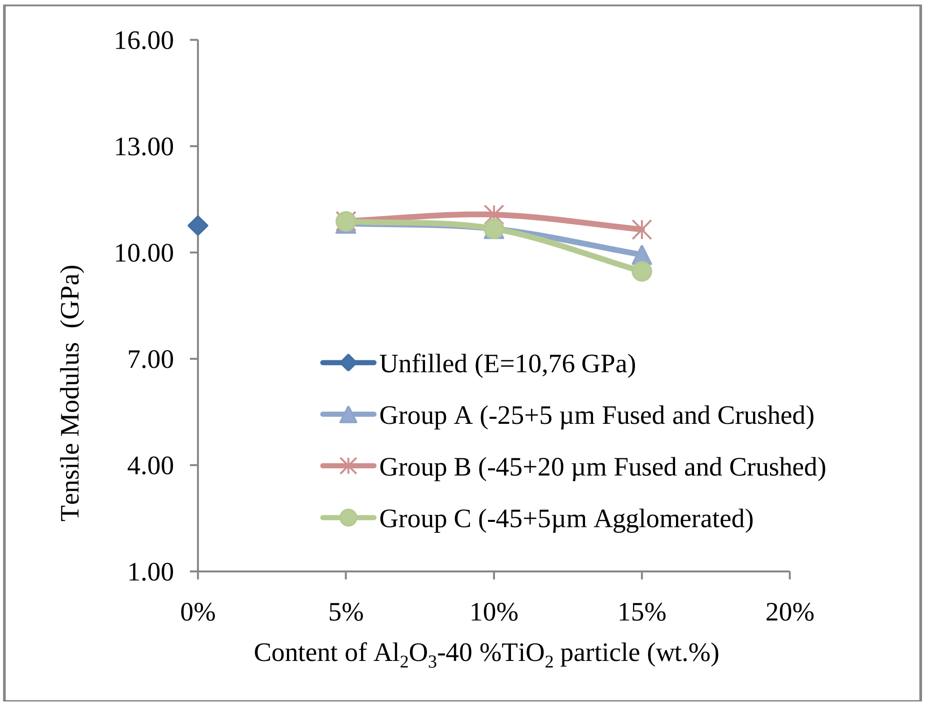


Figure 2. The variation of tensile modulus of the composites with weight fraction of particle content.

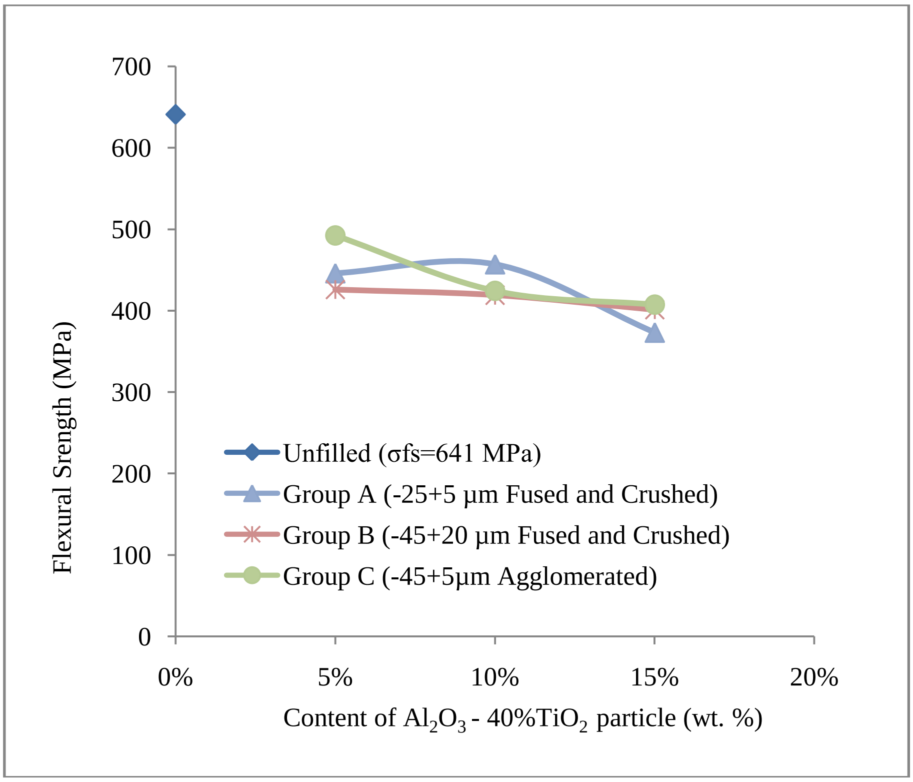


Figure 3. The variation of flexural strength of the composites with weight fraction of particle content.

In general, as the amount of ceramic powder filled into the composite material increases, the flexural strength values of the composite material decrease. However, an increase in the flexural strength values of the material at the 10% filler ratio in the ceramic powder reinforced materials in Group B has been observed. Among the ceramic powder filled composite materials, the highest flexural strength values were found in materials with a contribution rate of 5 wt.% in Group C. Because of the spherical structure of the agglomerated geometry of the ceramic powders in Group C, the stress concentration that causes crack initiation and propagation in the powder surroundings is lower [9]. When the flexural strength values of the same powder type materials are compared, the flexural strength of Group A samples with low powder particle size is higher than the flexural strength of Group B samples.

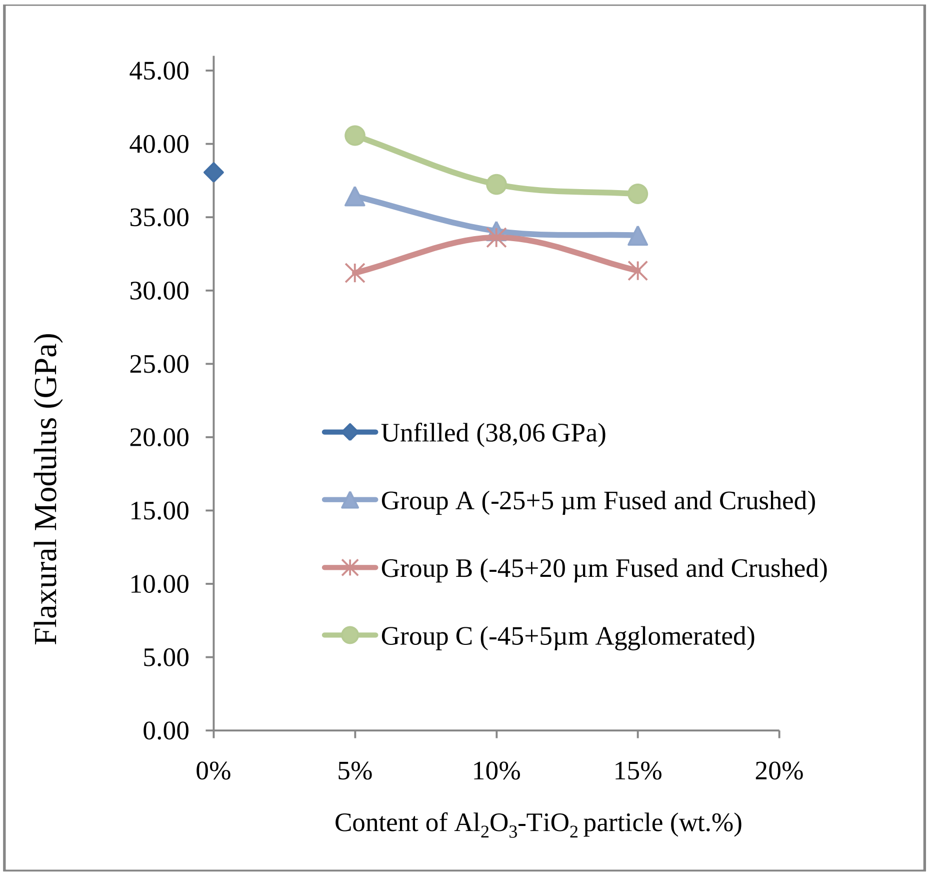


Figure 4. The variation of and flexural modulus of the composites with weight fraction of particle content.

According to the results of the flexural tests of composite materials; the flexural modulus of the composite materials with 5 wt.% ceramic powder added in Group C is the highest. Generally, as the ceramic powder reinforcement ratio added into the composite material increases, the flexural modulus values of the composite material decrease. However, an increase in the flexural modulus values of the material at the 10 wt.% filler ratio in the ceramic powder filled materials in Group B has occurred. When the flexural modulus values of the same powder type are compared, the flexural modulus values of Group A samples with low powder particle size are higher than the flexural modulus values of Group B samples.

As for flexural failure deflection of specimens under maximum flexural loading, it was found that all ceramic powder filled glass fiber reinforced epoxy composites have lower flexural failure deflection than the unfilled glass fiber reinforced epoxy matrix composite material. The flexural failure deflection is inversely proportional to the filler weight fraction and filler stiffness.

The interface bonding or adhesions between the filler and the matrix and the homogeneous filler dispersion have a great effect on the mechanical properties of particulate filled composite materials. Furthermore, the presence of agglomeration in the matrix is to be taken into consideration. Agglomeration can enhance flow characteristics of powders, which leads to poor packing and porous composites. The presence of agglomeration and voids in the composites obviously deteriorates their mechanical properties [8, 9].

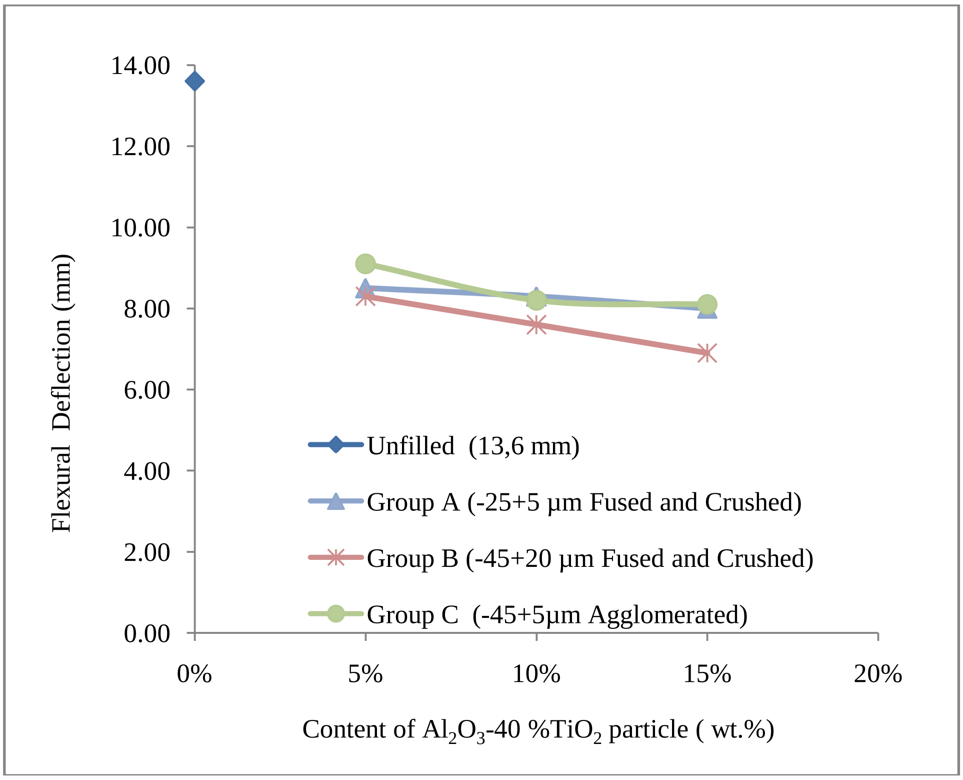


Figure 5. The variation of flexural deflections of the composites with weight fraction of particle content.

Also, high filler content leads to difficulty in mechanical stirring process and hence uniform distribution of filler in the composite laminate produced cannot be ensured. Strong interfacial bonding between the fiber and matrix contributes higher flexural properties. The increase of the tensile modulus of the glass fibre reinforced epoxy composites filled with Al_2O_3 – 40 wt. % TiO_2 particle may be attributed to the rigid nature of the fillers. At higher weight fractions of Al_2O_3 – 40 wt. % TiO_2 particle, the poor interface bonding or adhesion between the filler and the epoxy resin matrix, or the presence of a large agglomerate phase in the matrix may be occurred to cause the lower flexural properties of composite materials [9].

IV. Conclusion

In this study, an experimental investigation has been conducted to evaluate the effect of ceramic particle size and shape on the flexural properties of glass fibre reinforced epoxy matrix filled with different shape, size and proportions of Al_2O_3 – wt. 40 TiO_2 particles.

The following main conclusions can be drawn from this study:

1. The density and porosity values of the composite materials increased effectively with increasing the Al_2O_3 – 40 wt. % TiO_2 particle content.
2. The tensile strengths of the glass fibre reinforced epoxy resin composites decreased effectively with increasing the Al_2O_3 – 40 wt. % TiO_2 particle content.
3. The tensile modulus of the glass fibre reinforced epoxy composites first increased with increasing Al_2O_3 – 40 wt. % TiO_2 particles content and then decreased with further increase in Al_2O_3 – 40 wt. % TiO_2 particles content.
4. The particle weight fractions, sizes and geometries of the ceramic powders added into the resin matrix during the production of glass fiber reinforced epoxy matrix composite materials significantly affect the flexural properties of the composite materials.
5. The flexural properties of agglomerated ceramic powder filled composite materials are higher than the flexural properties of fused and crushed ceramic powder filled composite materials.

Acknowledgements

This work was financial supported by Research Fund of Uşak University. (Project Number:2014/TP011)

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