A Study of Mechanical Properties of Poly Methacrylate Polymer Reinforced by Silica Particles (Sio_2)

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ABSTRACT

In this research the mechanical properties of PMMA polymer reinforced by ceramic particles (silica) has been investigated. Many tests are performed on these composites. The effects of the particles size and volume fraction on the mechanical properties which include: ultimate tensile strength, elongation percentage, modulus of elasticity, bending modulus, flexural strength, max. shear stress, impact strength and fracture toughness were studied.

Statistical and mathematical analyses were used to the processing of the experimental data. Mathematical models were done which show the mechanical properties of composite materials as a function of particles size and volume fraction.

The results had revealed that the values of modulus of elasticity, elongation percentage, tensile strength, bending modulus and max. shear stress increase with the addition of SiO_2 particles and with the increase of the volume fraction of them and its reach the maximum value at (12% vol.) and (25 μm) particles size. The values of fracture toughness and impact energy decrease with increase of volume fraction. Silica particles with small particles size improved these properties more than that of large particles size.

Keywords: Mechanical properties, PMMA, Silica, Particles size, Volume fraction**.**

دراسة الخصائص الميكانيكية للبولي مثيل ميثااكريليت المقوى بدقائق السليكا (SiO_2)

الخلاصة

في هذا البحث تمت دراسة الخواص الميكانيكية لبوليمر البولي مثيل ميثااكريليك المقوى بدقائق سيراميكية (السليكا). اجريت للمادة المتراكبة العديد من الفحوصات وتم دراسة تاثير الحجم الحبيبي والكسر الحجمي على الخواص الميكانيكية المتمثلة بمعامل المرونة ومقاومة الشد والأستطالة % ومقاومة الانحناء وأقصى أجهاد قص ومقاومة الصدمة ومتانة الكسر.

واستخدمت تحليلات احصائية و رياضية لمعالجة النتائج العملية وتم عمل نماذج رياضية تبين الخواص الميكانيكية كدالة للحجم الحبيبي والكسر الحجمي.
بينت النتائج ان قيمة معامل المرونة ومقاومة الشد والاستطالة % ومعامل مرونة الانحناء ومقاومة الانحناء وأقصى اجهاد قص تزداد مع زيادة نسبة SiO_2 ومع زيادة الكسر الحجمي لهذه الدقائق وصلت الى قيمتها العظمى عند(12% vol) كسر حجمي و (25 µm) حجم حبيبي.
اما متانة الكسر ومقاومة الصدمة فانها تقل مع زيادة الكسر الحجمي للسيلكا وان دقائق السيلكا ذات الاحجام الحبيبية الصغيرة تحسن الخواص اكثر من الدقائق الكبيرة الحجم.

NOTATION

Symbol	Meaning	Unit	Symbol	Meaning	Unit
A	Cross sectional area	m^2	D**L**	Elongation	m
a_o, a_1, a_2, a_3, a_4, a_5	Regression coefficients	-	**P**	Load	N
b	Width of specimen	M	**R**	coefficient of multiple determination	-
d	Thickness of specimen	M	SSE	Sum of squared error	-
E	Modulus of elasticity	MPa.	SSR	Regression sum of squares.	-
E_b	Modulus of elasticity in bending	MPa.	SST	total correct sum of squares	-
e	Error of the response	-	**U_c**	Impact energy	J
F	Applied load	N	**X**	Volume fraction	%
F.S	Flexural strength	MPa	**Y**	Response	-
G_c	Toughness of material	J/m^2	**Z**	Particles size	µm
I	Moment of inertia	m^4	s	Stress	MPa
K	properties	-	e	Strain	-
k_c	Fracture toughness	$MPa.m^{0.5}$	d	Deflection of specimen	m
L	Length of specimen	M	$\tau_{max.}$	Max. shear stress	MPa
L_o	Original length	M	x_1, x_2, x_3	Independent variables	-

INTRODUCTION

A Composite is a multiphase material that is artificially made and chemically dissimilar and separated by distinct interface. One of these phases is termed the matrix which is continuous and surrounds the other phase often called the reinforcement phase which consists of three main divisions: particles, fibers and structural, which should be much stiffer and stronger than the matrix. The mechanical properties can be varied substantially through changes in the microstructure, the morphology, volume (or weight) fraction and mechanical properties of the reinforcing phase, and the nature of the interface between matrix and the reinforcement [1 & 2].

Many studies had investigated the effect of volume fraction, type and particle size of particles on mechanical properties of composite materials.

kinson [3] 2000, studied the particle size effect of carbon black on rubber composite. It was found that the tensile, tear strength and abrasion resistance increases with decreasing particle size of carbon black due to the possibility of occurring the chemical cross linkages in the carbon-rubber interface.

A. K Pogosyan et al. [4] 2002 studied the influence of type, shape, volume content and particle sizes of the fillers on the tribogical properties of polymers composite. The results demonstrated the best improvement in wear resistance of the epoxy was at a nano-particles content of 4 Vol % TiO_2.

H. Kereem [5] 2002, studied the mechanical properties of epoxy reinforced by nickel particles and has been used various sizes with different volume fraction of particles. The results found that the values of modulus of elasticity and yield strength increase with the increase of the volume fraction of particles, as well as increase of the size particles have improved properties and up (32µm).

Marur et al. [6] 2004, studied the influence of the particle size and volume fraction of submicron spherical alumina particle in an epoxy matrix on the fracture toughness of the composite, three particle size, 50 nm, 500 nm and 5 mm, are used. It is found that particle size significantly affect the static and dynamic fracture toughness and was found when the particles size is (5 µm) the strength increases with increase of the weight fraction of particles.

Malachy et al, [7] 2006, studied the mechanical properties (tensile strength, compression strength and impact energy) of groundnut husk powder filled with polyurethane composite. Filler content was varied between 2 and 10 weight percent of matrix. It was observed that the tensile strength increases marginally with increase in filler content, while the impact strength decreases with increased filler loading. These effects are attributed to the poor interfacial interactions between reinforcement and matrix.

Suvangshu et al, [8] 2009, investigated the polyurethane nanocomposites with different weight percentage of clay loading (1 %, 2.5 % and 5 %) have been evaluated as biocompatible materials. The mechanical properties, such as tensile strength and scratch hardness, were improved 2 and 5 times, respectively by nano composite formation. Even the impact resistance improved a little.

N.J. Shaleh and S. N. Mustafa [9] 2011, studied some of mechanical, thermal and physical properties of polymer blend mixed with different weight percentage and different particle sizes of Iraqi kaolin filler, it was found that most mechanical properties such as hardness, modulus of elasticity and compression increase while impact and elongation decrease with increasing weight percentage and a decreasing particle size.

The aim of this work is to study the effect of the volume fraction and the particles size of (SiO_2) particles on the mechanical characteristics of the Polymethyl methacrylate. And make mathematical modeling of the property of the experimental results representing the effect of the (volume fraction and particles size) on the properties.

THEORETICAL PART

Tensile test (Stress-Strain)

Tensile test is widely used to provide the designer with information about material strength and maximum elongation and others. The stress used in such curve is a longitudinal stress in test specimen and expressed as:

$$\sigma = E.\varepsilon \qquad \dots (1)$$

Where:

$\sigma = P/A$ and $\varepsilon = \Delta L/L$

Tensile strength, modulus of elasticity and elongation percentage can be calculated from the tensile curve [10].

Impact Strength test

The impact properties of a material represent its capacity to absorb and dissipate energies used to measure the strength of material under impact or shock loading [11].

Izod and Charpy tests are ordinarily conducted to assess impact strength or by falling weight impact test.

Impact strength is calculated from the following equation [2]:-

$$G_c = \frac{U_c}{A} \qquad \dots (2)$$

Fracture toughness, which describes the ability of a material containing a crack, to resist fracture, can be expressed as [8]:-

$$K_c = \sqrt{G_c \cdot E} \qquad \dots (3)$$

Flexural strength test

In engineering mechanics, flexure (or bending) characterizes the behavior of a structural element subjected to an external load applied perpendicular to the axis of the element.

The maximum flexural strength in this test can be calculated by the following equation [12]:-

$$F.S = \frac{3 \cdot P \cdot L}{2 \cdot b \cdot d^2} \qquad \dots (4)$$

The bending modulus (E_{Bend}) can be determined by using Equation (5)

$$E_{Bend} = \frac{f \cdot l^3}{48 \cdot I \cdot d} \qquad \dots (5)$$

The moment of inertia of specimen is determined using Equation (6)

$$I = \frac{b \cdot d^3}{12} \qquad \dots (6)$$

Also the maximum load at failure, the max. shear stress is determined using Equation (7)

$$t_{\max.} = \frac{3 \cdot P}{4 \cdot b \cdot d} \qquad \dots (7)$$

Mathematical Model

The mathematical and statistical techniques are used for modeling analysis of the process in which the response of interest is influenced by several variables and objectives.

In many engineering fields, there is a relationship between an out put variable of interest (y) and a set of controllable variables (x_1, x_2, …x_n) which represent the model in the form.

$$Y=f(X_1, X_2,\dots\dots\dots X_n) + e \qquad ...(8)$$

The statistical analysis of the developed mathematical models is done by statistical package for the social sciences (SPSS) by using the response surface modeling.

Response surface methodology (RSM) is a very practical, economical and an effective statistical tool and widely used in experimental design, model fitting, analysis of the experimental results, validation and condition of optimization [13].

In this study, multiple polynomial (least square fitting) regression analysis is used to establish a mathematical model among the experimentally obtained parameters. Multiple regression analysis techniques are applied to relate the property to volume fraction and particles size of SiO_2, the best form of the relationship between the property, particles size and volume fraction of SiO_2 parameters is chosen in the form of [14]:

$$K = a_o + a_1 \cdot X + a_2 \cdot Z + a_3 \cdot X^2 + a_4 \cdot Z^2 + a_5 \cdot X.Z \qquad \dots (9)$$

The analysis of variance (ANOVA) is also called coefficient of multiple determination referred to as (R) measuring the proportionate reduction of total variation in association with the use of the set of predictors in the model (is used to cheek the validity of the model).

It is defined in terms of SST, SSR, and SSE as:

$$R^2 = \frac{SSR}{SST} = 1 - \frac{SSE}{SST} \qquad ... (10)$$

EXPERIMENTAL WORK

Properties of Used Materials

- **Poly Methyl Methacrylates**

These are thermoplastic polymers of alkyl acrylates. Acrylic resins have good optical clarity, weatherability, surface hardness, chemical resistance, rigidity, impact strength, and dimensional stability [16]. The properties of PMMA are given in Table (1).

Table (1) Properties of PMMA Polymer [16].

Properties		Conditions	
		State	ASTM
Elastic Modulus (MPa)	2553 - 3174	compressive	D638
	2243 - 3243	tensile	
Tensile Strength (MPa)	48 - 72	at break	
	54 - 73	at yield	
Elongation at break (%)	2 - 6		
Flexural Modulus (MPa)	2243 - 3174	23 ºC	D790
Flexural Strength (MPa)	72 - 131	at yield or break	

- ***Silica particles***

Silica has considerably low thermal expansion, fairly high melting point and is resistance to creep making it good refractory material [17].

Physical, mechanical, thermal and electrical properties of quartz are gives in Table (2).

Table (2) some properties of quartz [18].

Property of Quartz	Value
Density (g/cm^3)	2.65
Tensile strength (MPa)	55
Compressive strength (MPa)	2070
Poisson's ratio	0.17
Fracture toughness (MPa)	-
Modulus of elasticity (GPa)	70

Preparation of the Specimens

The composite specimens were made from (Poly methyl methacrylate) as a matrix and silica (SiO_2) as the reinforcement with different volume fractions of (3%, 6%, 9% and 12%) and five different particles sizes (25 µm, 53 µm, 75 µm, 106 µm , and 212 µm) by mixing the ceramic particles in liquid Polymethyl methacrylate followed by pouring in an open mould. The mixture was left in the mould for (24) hrs at room temperature to solidify. Then the samples were placed in an oven for 1 hr at 55°C. This step was important to reveal complete polymerization, best coherency, and to relieve residual stresses. The specimens were cut according to the standard dimensions for each test, using different cutting tools.

MECHANICAL TESTS

Tensile Test

This test is performed according to (ASTM D638M- 87b) at room temperature [10]. The used tensile machine is (INSTRON 1195 50 KN, made in England). Figure (1) shows a standard specimen for tensile test.

Impact Test

This test is performed according to (ISO-180) at room temperature [11]. Izod impact test machine used for testing polymeric materials and the of the impact strength and fracture toughness values depended on the calculation of the required energy for fracture. Figure (2) shows a standard specimen for impact test.

Flexural Strength Test

This test is performed according to (ASTM D790) at room temperature [12]. Figure (3) shows a standard specimen for bending test**.**

RESULTS AND DISCUSSION

Tensile Test

- Modulus of Elasticity

Figure (4) shows the relationship between the modulus of elasticity and the volume fraction of the SiO_2 particles, which were added to the PMMA resin. It can be noted that the modulus of elasticity increases with increasing volume fraction. So, the volume fraction of (12 %Vol.) represents the greatest value for the modulus of elasticity (1.177 Gpa) for PMMA reinforced with SiO_2 particles with particles size of (25 µm). This may be due to the strengthening mechanism and the nature of bonding mentioned before. Also, the increase in elastic modulus of the composite material may be due to the fact the elastic modulus of SiO_2 range is (70 Gpa) and it is much higher than that for PMMA resin [19].

From this figure, also it can be seen that small particles of SiO_2 reinforcing particles improve the modulus of elasticity more than large particles of SiO_2 reinforcing fillers, this is due to the strengthening mechanism as follows ***firstly***: large particles strengthening for SiO_2 reinforcing particles tend to restrain the movement of the matrix phase in the vicinity of each particles, whereas the matrix transfers some of the applied load to the particles and bear fraction of it, and ***secondly***: the small particles hinder or impede slipping of matrix chains and require high stress to bow them in narrow space among particles compared with large particles of SiO_2 and the matrix bears the major portion of the applied load [20].

- Ultimate Tensile Strength

From Figure(5), it can be seen that SiO_2 reinforcing particles improve ultimate tensile strength, and it increases with increase in the volume fraction for all particles sizes of SiO_2, due to the power of high linkage between the matrix material and reinforcement material, which leads to reducing the slip during the tension.

It can be seen clearly that small particle size has a noticeable effect on the tensile strength more than the large particles due to the same reason mentioned before, so that the ultimate tensile strength at volume fraction of (12%) of (25 µm) particles size reaches (36 MPa), while (34, 31.3, 30, and 27.5 MPa) are at (53 µm, 75 µm, 106 µm, and 212 µm) respectively.

- Elongation Percentages at Fracture

Figure (6) shows the relationship between the elongation percentage calculated at fracture point and the volume fraction of the SiO_2 particles of (25 µm, 53 µm, 75 µm, 106 µm, and 212 µm), which were added to the PolyMethyl Methacrylate resin, respectively. It could be observed from figure (6), that the elongation percentage at break increases with the increase in volume fraction of reinforcing SiO_2. This is due to the second role of reinforcing, as well as reinforcement role, which is the voids filling that naturally exist in polymer structure, then they diminish these discontinuous regions which act as stress raiser therefore break strength and strain (elongation) increase.

Also can be noted from this figure that the finer particles size increase the elongation percentage at break more than larger particles size of SiO_2 reinforcing particles.

Bending Test

- bending modulus

Figure (7) illustrates the bending modulus that increases with increasing volume fraction of (SiO_2) particles, and reaches its maximum amount of (5120) Mpa at addition volume fraction of (12% vol.) and (25 µm) particles size compared to the bending modulus of the reference (PMMA) which is equal to (2101 Mpa). The increase in bending modulus may be also due to the high elastic modulus of the SiO_2 compared with that of the matrix material [19].

- Flexural strength

Figure (8) shows the relationship between the bending strength (flexural strength) and the volume fraction of the fillers particles of SiO_2 powder, which were added to the PMMA resin.

The figure illustrates that the bending strength increases with increasing volume fraction of SiO_2 filler particles, and reaches its maximum amount at 12 % vol at (25 µm) which is (355.19 Mpa). This can be related to the strengthening mechanism and the nature of the bonding between the matrix and reinforcement. The increase in bending strength may be also due to the high elastic modulus of the reinforcing material compared with that of the matrix material.

- Max. shear stress

Figure (9) shows the max. shear stress of composites used in this study. The maximum shear stress is obtained in (12 %) volume fraction and (25 µm) particle size of SiO_2 particles. The highly (reinforcing- matrix) bonding of the composites has a remarkable effect on increasing the max. shear stress. The presence of SiO_2 particles with PMMA matrix increases the max. shear stress due to ability of these particles to hinder the crack propagation in addition to the strong bonding of PMMA with the reinforcement [21].

It can also be seen that the small particles of reinforced filler can improved the bending properties more than large particles. This is due to the SiO_2 particles with fine particles size provided composite material with high strong bonding which were mentioned before that effect strongly on the bending properties of the composite material.

Impact Test

- Impact strength

Figure (10) shows the relationship between the impact strength and the volume fraction of the SiO_2 particles, which were added to PMMA resin.

This figure illustrates that the impact strength decreases with increasing volume fraction of SiO_2 particles of composite materials. This is because of the SiO_2 particles, which may represent points for a localized stress concentration, from which the failure will begin, so that the composite tends to form a weak structure. Also, an increase in concentration of particles reduces the ability of matrix to absorb energy, thereby reducing the toughness, so impact energy decreases.

- fracture toughness

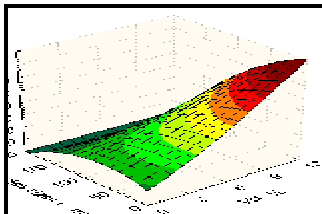
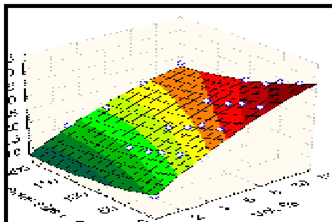
Figure (11) shows the relationship between the fracture toughness and the volume fraction of the SiO_2 particles added to the PMMA resin. The figure illustrates that fracture toughness decrease with increasing volume fraction of SiO_2 particles, and reaches its minimum amount at (12 vol %). This is due to the dependence of the fracture toughness on the elastic modulus, as mentioned before, and the impact strength shown a similar behavior to the behavior of the fracture toughness as explained. Also, the composite material filled with small SiO_2 particles has higher fracture toughness than the other which was filled with large SiO_2 particles, according to the impact strength of the small particle composite material, which is higher than that for the large particle composite material.

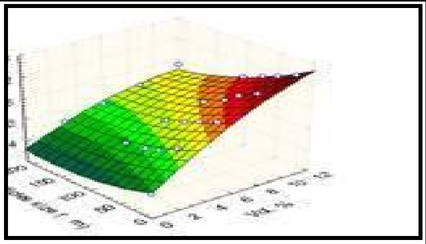
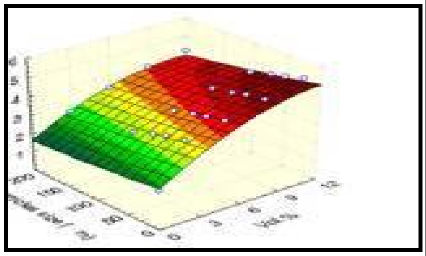
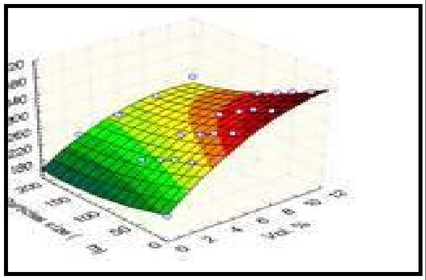
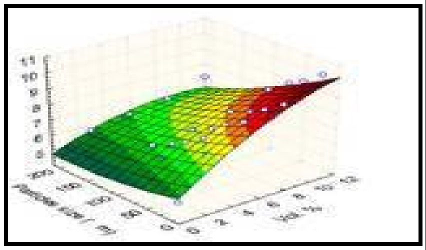
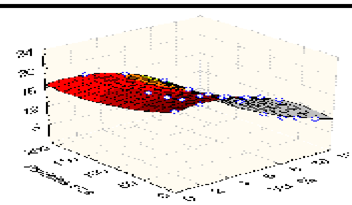
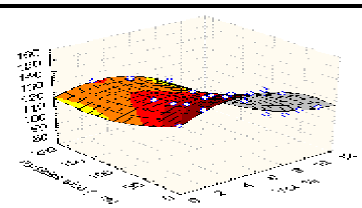
Mathematical Model

It can be seen from these models that the volume fractions have greater effect than the particle size on the properties.

The final model coefficient of multiple determinations (R^2) of the properties as function of (X = volume fraction of SiO_2) and (Z=particles size of SiO_2) at a given working conditions for each test is represented by the equations in Table (3).

Table (3) Mathematical Model of Properties.

Property	Equation of mathematical model and coefficient of multiple etermination	Figure
Modulus of elasticity (Gpa)	E=865.682+26.30313X-0.6066 Z+0.13864 X^2 +0.002089Z^2-0.08 .X.Z R^2=0.93	
Tensile strength (Mpa)	T=13.9168+2.87352X-0.0671Z-0.07261X^2 +0.00018Z^2-0.00216.X.Z R^2=0.96	

at break %	$\varepsilon=1.52326+0.25289X-0.00574Z-0.0065X^{2}+0.00002Z^{2}-0.00037.X.Z$ $R^2=0.92$	
Bending modulus (Mpa)	$E_B=2195.85+473.53X-3.835Z-16.2X^{2}+0.0057Z^{2}-0.2125.X.Z$ $R^2=0.96$	
Flexural strength (Mpa)	$\sigma=204.385+25.866X-0.50148Z-0.96X^{2}+0.0014Z^{2}-0.0277.X.Z$ $R^2=0.97$	
Max.shear stress (Mpa)	$\tau=5.6342+0.60324X-0.00979Z-0.02X^{2}+0.00002Z^{2}-0.001.X.Z$ $R^2=0.88$	
Impact strength (KJ/ m^2)	$I=20.7936+0.45576X-0.04551Z-0.106X^{2}+0.00012Z^{2}-0.00024.X.Z$ $R^2=0.9$	
Fracture toughness (Mpa.$\sqrt[2]{m}$)	$F=133.611+5.53993X-0.2467Z-0.59X^{2}+0.00076Z^{2}-0.00717.X.Z$ $R^2=0.92$	

CONCLUSIONS

The conclusions are:

1. The addition of the reinforcing particles (SiO_2) to the PMMA polymer leads to improve the mechanical and physical properties.
2. Ultimate tensile strength, modulus of elasticity and elongation percentages at break increases with the increase of the volume fraction of the reinforcing particles SiO_2 for the PMMA polymer. The small SiO_2 particles improved the tensile properties of pure PMMA more than that of large SiO_2 particles.
3. The largest values of ultimate tensile strength, modulus of elasticity and elongation percentages at fracture were at (12 % vol) of SiO_2 reinforcing particles.
4. Bending modulus, flexural strength and max. shear stress increases with increasing volume fraction of (SiO_2) particles and reaches its maximum value at addition volume fraction of (12% vol.) and (25 µm) particle size of SiO_2.
5. The impact strength and fracture toughness for the prepared composite materials decreases with increasing volume fraction of SiO_2 particles.
6. Mathematical models represent the behavior of properties of composite materials as a function of particles size and volume fraction.

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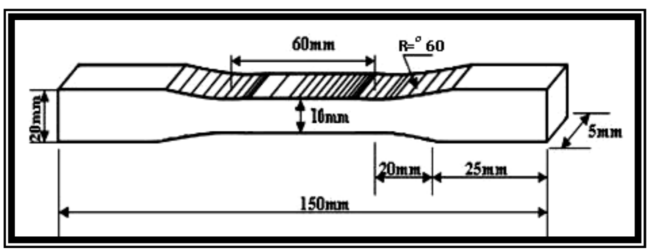
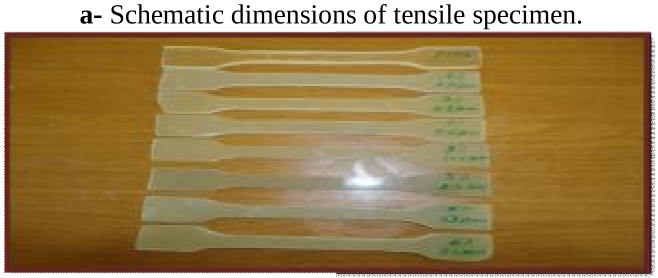
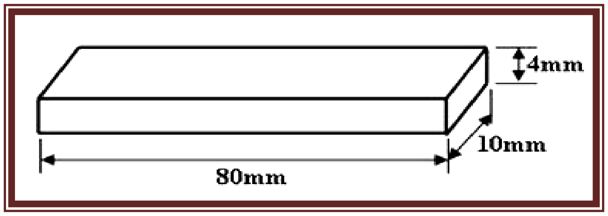


a- Schematic dimensions of tensile specimen.



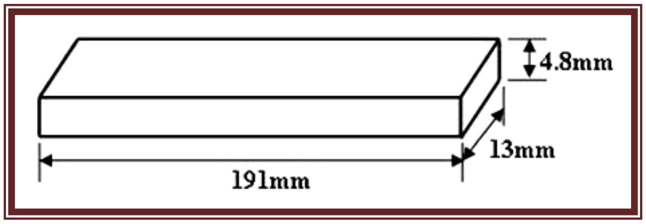
b- Experimental specimens.
Figure (1) Tensile test specimens.



a. Schematic dimensions of impact specimen.

b. Experimental specimens.

Figure (2) Impact test specimens.



a- Schematic dimensions of bending specimen.

b- Experimental specimens.

Figure (3) Bending test specimens.

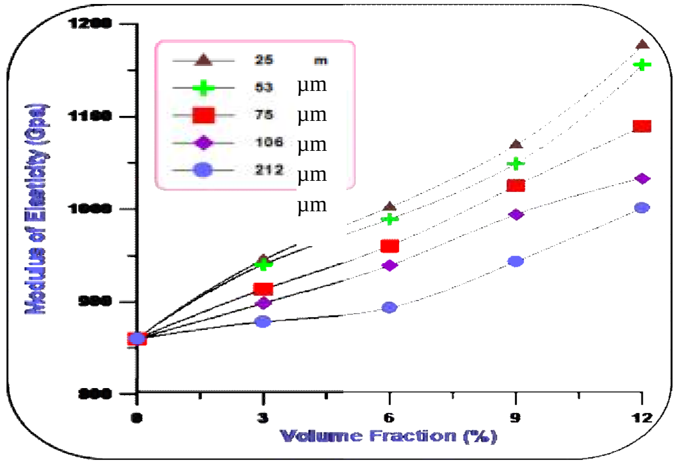


Figure (4) The relationship between the modulus of elasticity and volume fraction for PMMA reinforced with SiO_2 particles.

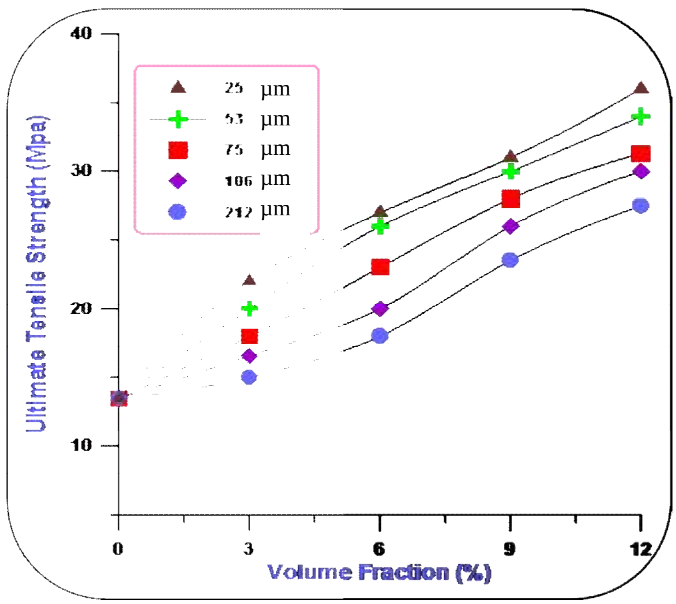


Figure (5) The relationship between the tensile strength and volume fraction for PMMA reinforced with SiO_2 particles.

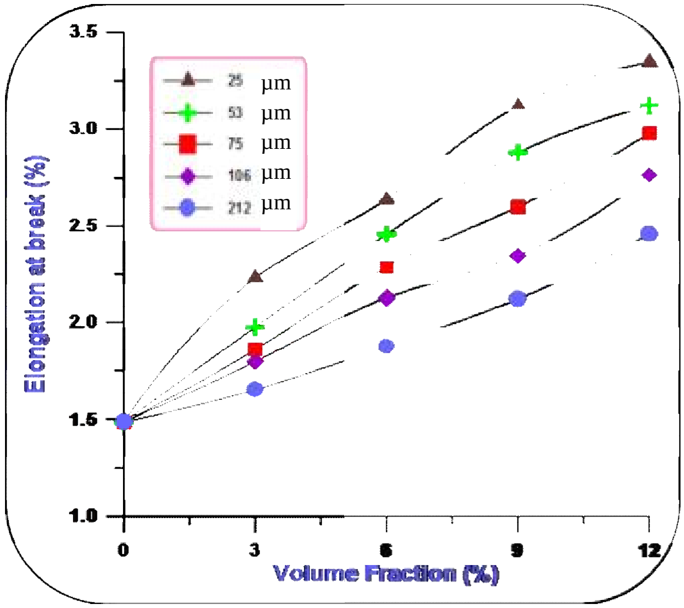


Figure (6) the relationship between the elongation percentages at break and volume fraction for PMMA Reinforced with SiO_2 particles.

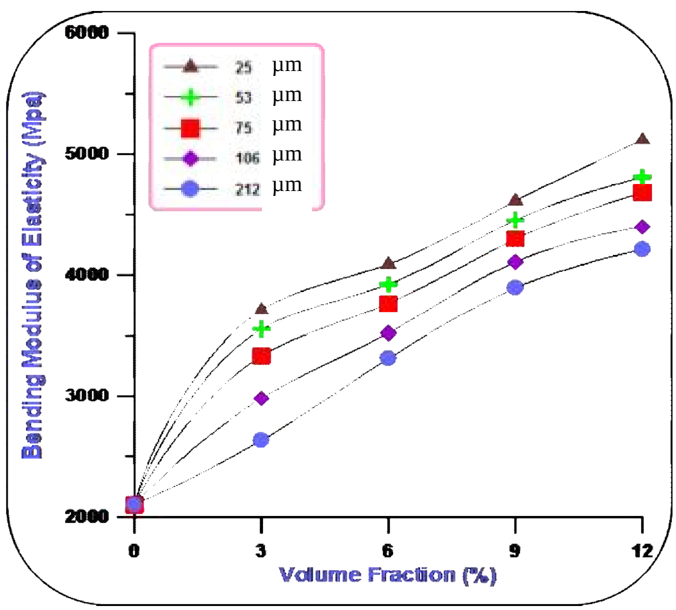


Figure (7) The relationship between the bending modulus of elasticity and the volume fraction for PMMA reinforced with SiO_2 particles.

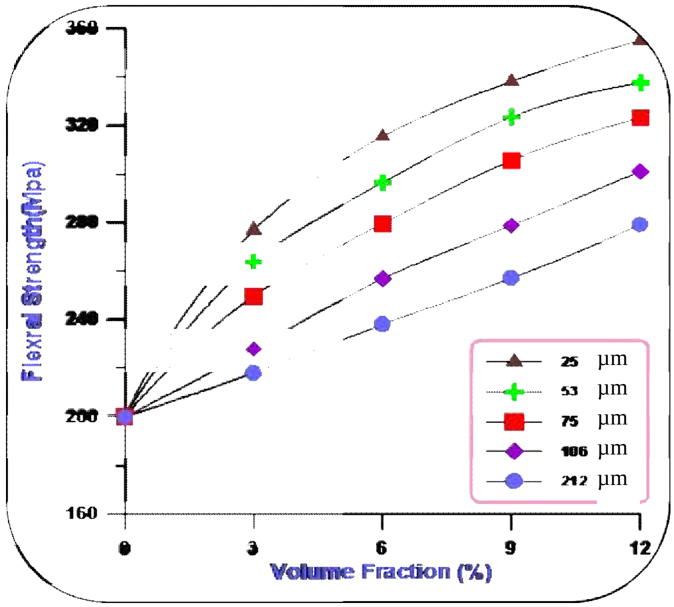


Figure (8) The relationship between the flexural strength and the volume fraction for PMMA reinforced with SiO_2 particles.

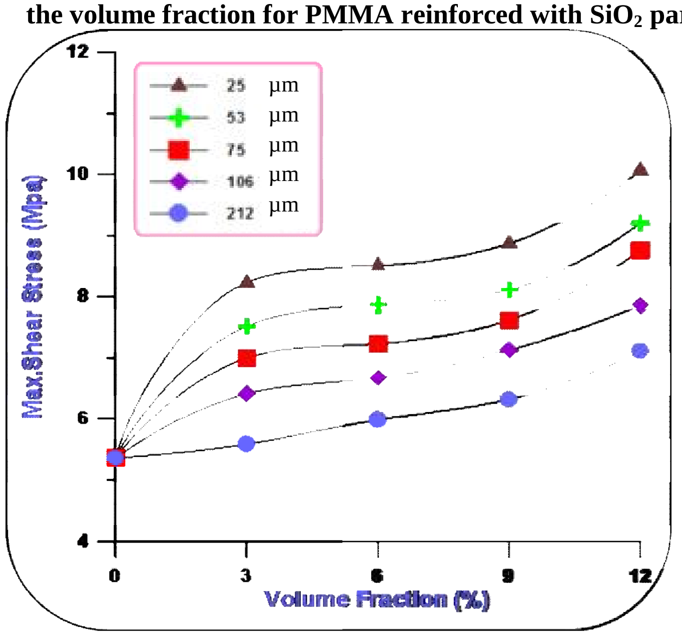


Figure (9) The relationship between the max.shear stress and the volume fraction for PMMA reinforced with SiO_2 particles.

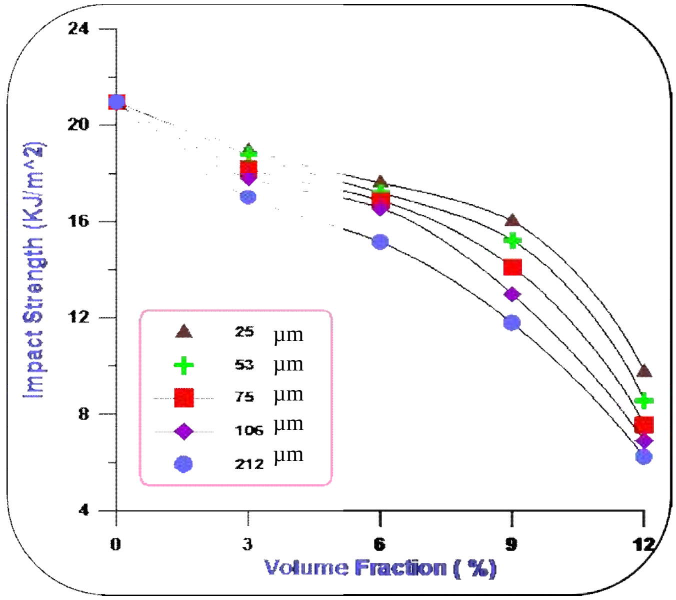


Figure (10) the relationship between the impact strength and volume fraction for PMMA reinforced with SiO_2 particles.

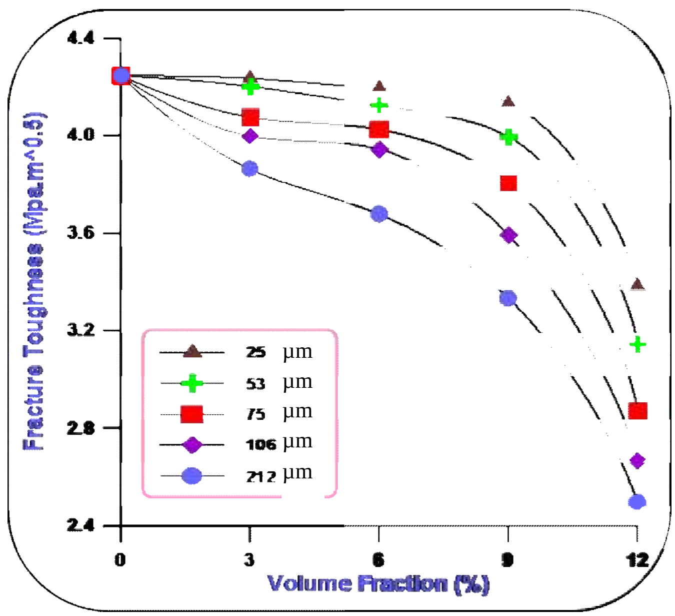


Figure (11) The relationship between the fracture toughness and volume fraction for PMMA reinforced with SiO_2 particles.