

Quartz Reinforced Unsaturated Polyester Resin Composites: Preparation and Characterization

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Authors' contributions

This work was carried out in collaboration among all authors. Author SKD designed and conduct the research work author MRN performed the statistical analysis, conduct and drafted the manuscript. Author RCD helps to design the specific part of the manuscript, authors MM and SB managed the literature searches and conduct specific experiments. All authors read and approved the final manuscript.

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ABSTRACT.

Compression molding has produced quartz-reinforced polyester composites (QPCs) weighing 10 to 40 per cent quartz relative to the weight of unsaturated polyester resin. Synergistic changes were made in the composite properties and were superior to those of the individual components. The composite's physical and mechanical properties such as bulk density, water absorption, tensile strength, flexural strength, hardness have illustrated the competency of the composite being developed. It was found that for the resultant composite examined, the percentage of water absorption is very small. However, when quartz content were increased, water absorption grew very slowly. Enhancement of mechanical properties strongly corresponds to strong adhesion force of quartz with the matrix and it influenced by well-disperse quartz particles on the whole surface of composite. This paper also performed thermal characterization of the composites. Because of these remarkable properties, as prepared composite can find applications in packaging, fuel cell, solar cell, structural materials and households purposes.

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1. INTRODUCTION

In materials research, very rapid development of polymer matrix composites appears on the various multidisciplinary sciences which attract people to use it during the last decades. Composites are one of the most widely used materials that have high physical, thermal and mechanical properties compared with conventional materials due to their adaptability to different situations and the relative simplicity of combination with other materials to serve specific purposes and desirable properties [1]. However, composites are utilized as mirror housing on many fields include: packaging, fuel cell, solar cell, fuel tank, plastic containers, impellers and blades for vacuum cleaners, power tool housing, and cover for portable electronic equipment such as mobile phones and pagers [2]. In this scenario, a common approach is to produce composite materials by varying a range of matrix materials and reinforcing agents. Generally, various polymers such as: rubber, polylactic acid, polyvinyl alcohol, acrylic latex, polyethylene, and thermoplastic starch are suitable as matrix in these types of composite materials [3]. Moreover, using a variety of matrices, unsaturated polyester resin is most commonly used for the preparation of advanced composite materials due to its cost efficiency, ease of handling and fabricating complex parts with less tooling cost and also have excellent room temperature properties [4]. Therefore, polyesters were suitable for a variety of applications mostly used in reinforced plastics used in the marine and transportation industries [5,6].

In recent years, many researchers focused on the use of some fillers into a matrix. Fillers will be more advantageous as matrix reinforcement due to good interaction between filler-matrix and improve the appearance of composites [7]. Various materials have already been tested as fillers such as: aluminum powder, carbon fiber, graphite, calcium carbonate, silica, clay, precipitated calcium carbonate (PCC), kaolin, talc, quartz and carbon black. Furthermore, when compared to the activity of sand (which is made up of a variety of minerals such as quartz, feldspars, mica, and other silicate minerals) as a filler, quartz has the potential to improve strength, adjust viscosity, highly resistant to weathering and create a smooth surface due to the presence of a specific crystal form of SiO_2 .

[8]. The addition of quartz enhances the thermal, rheological, mechanical and adhesion properties of polyester due to the creation of hydrogen bonds between the groups of silanol on the quartz surface and the soft segments of ester carbonyl groups [9,10]. Profound studies have been done on fracture toughness, bending, and compression loading modes of quartz filled unsaturated polyester resin composites with a silane coupling agent and also the significance of coupling in highly filled particle composites [11]. It is therefore necessary to choose an effective reinforcement that can improve the physical, mechanical and thermal properties of the composite materials for different applications.

In the present study, the composites of the polymer matrix were developed where high strength quartz reinforcing was combined with a matrix of polymer (polyester resin). The composites were prepared by compression molding process and then characterized their properties such as physical, mechanical and thermal properties using appropriate instruments. The novelty of this study is to develop a quartz reinforced polyester composite and estimate its competency in the respect of physical, mechanical and thermal properties. Besides shorten steps in the synthesis process of quartz reinforced polyester composite for reducing overall cost during synthesis and produce high purity of products.

2. EXPERIMENTAL DETAILS

2.1 Materials

The materials needed for nanocomposite fabrication in this research work were unsaturated polyester resin, quartz, styrene monomer and methyl ethyl ketone per oxide (MEKP) as hardener. All the reagents were of commercial grade and purchased from the local market of Dhaka, Bangladesh.

2.2 Methods for Sample Preparation

In order to prepare the quartz-polyester composite, firstly quartz washed properly for a long time thus several waste washed away and then sieve analysis was taken. The average diameter for the individual quartz varied from 43 to 125 microns. They were then dried in a preheated oven at 100°C for 24 hours. According

to work plan quartz was taken from 10 to 40 wt % for my research experiment. Table 1 shows different composition of different samples.

In this case, required quantity of different percentages of quartz and polyester resin (with 10% styrene monomer) was mixed in a bowl very carefully with a continuous stirring for about half an hour. Ethyl methyl ketone peroxide was used as a hardener, as an amount of 1.5 wt % of polyester resin and styrene in the amount of 10 per cent of polyester resin. Then the mixer was poured into the mold and kept for drying to make quartz reinforced polyester composite. After 4 to 5 hours of drying the composite specimen was ready for further testing. These composite sides were then smoothed with polyethylene sheet and were characterized by different standard methods [12], [13]. Fig. 1 shows the four different quartz polymer composite samples getting browner with the increasing percentage of quartz.

2.3 Characterization

Four samples of quartz reinforced polyester composites having a composition of quartz to polyester in the ratio of 10:90, 20:80, 30:70, 40:60 were prepared and were characterized by

different standard methods. Different mechanical properties such as bulk density, water absorption, flexural properties, compressive strength, tensile properties and elongation at break were determined for these four types of composite by Universal testing machine (Model 1011UK, INSTRON Corporation).

For bulk density measurement, the specimen was prepared according to ASTM C135-76 [14]. Bulk density was calculated using following formula,

$$D = \frac{W_s}{V} \quad (1)$$

Where, D=Density of the specimen in kg/m³, W_s=Weight of the specimen in kg and V=Volume of the specimen in m³.

For water intake measurement, the test specimen was cut into 10 to 12 mm long, 5 to 6 mm broad, and 3 to 4 mm high dimensions in accordance with ASTM D1505-18 [15]. The sample was then immersed in distilled water (23^o C) for roughly 96 hours in order to measure water absorption according to ASTM D570-98 [16]. Every 24 hours, the percentage of water absorption was measured.

Table1. Composition of quartz- polyester resin composite

Composite	Quartz powder (%)	Polyester resin (%) (with 10% styrene monomer)
Sample-1	10	90
Sample-2	20	80
Sample-3	30	70
Sample-4	40	60

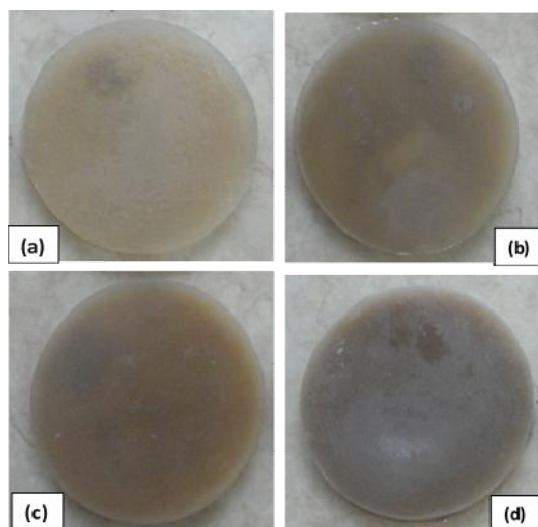


Fig. 1. Quartz-polyester composite- (a) 10% Quartz (b) 20% Quartz (c) 30% Quartz (d) 40% Quartz

Total water absorption was calculated using following formula:

$$\% \text{ water absorption} = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Dry Weight}} \times 100\% \quad (2)$$

The flexural strength was carried out by using universal testing machine (model 1011 UK, INSTRON Corporation). In this experiment, the support span length was 45 mm and the test speed was 5 mm/min. The test specimen was fabricated in accordance with ASTM D 790-02 [17]. The ASTM D790-17 standard method [18] was used to conduct the test by using the following equation.

$$S = \frac{3PL}{2BD^2} \quad (3)$$

Where, S = Stress in the outer fibers at mid span, Mpa. P = Load at a given point on load – deflection curve, N. L = Support span, mm. B = Width of specimen tested, mm. D = Depth of tested specimen, mm.

Tensile strength test was carried out according to the standard method described in ASTM D638-14 [19] and specimen was prepared accordance with ASTM D638-02a [20].

Tensile strength can be calculated by using following equation:

$$\text{Tensile strength} = \frac{\text{Applied load}}{\text{Cross sectional area of the load bearing area object}} \quad (4)$$

Percent Elongation (PE) is defined as the ratio of maximum elongation to original gage length and is given with tensile strength, which is a measure of ductility.

$$\frac{\text{Percent Elongation}}{\frac{\text{Final gage length} - \text{Original gage length}}{\text{Original gage length}}} \times 100 \quad (5)$$

The rebound Hardness was measured by Leeb Rebound Hardness Tester (Model H1000). Rebound hardness of the as prepared composites was calculated by using the following equation [21].

$$HL = \frac{100 V_I}{V_R} \quad (6)$$

Where, HL is the rebound hardness; V_I is the velocity of the indenter before impact; V_R is the velocity of the indenter after the impact.

The thermo-gravimetric analysis was carried out on a TGA / DTA Machine (TG/DTA 6300), which is capable of simultaneously performing Thermogravimetric (TG), Differential Thermal

Analysis (DTA), and Differential Thermogravimetric (DTG).

Thermo mechanical analysis (TMA) is a suitable device for determining various thermal properties of composite materials, which involves measuring the sample's dimension change as a function of temperature, time or force. This sort of analyzer is mostly used to calculate thermal expansion coefficients, but it can also be used to calculate the glass transition temperature. Thermomechanical analysis (TMA) of the composites were determined by TMA/SS 6300 system, Seiko Instrument, Inc. Japan.

The coefficient of thermal expansion (α) was calculated using the equation [22].

$$\alpha = \frac{\Delta L}{L \Delta T} \quad (7)$$

here, L=Initial Length of sample

3. RESULTS AND DISCUSSION

The bulk density effect of variation of wt.% of quartz on the bulk density of quartz reinforced polyester composites were investigated in Fig.2. Varying the amount of quartz (10%, 20%, 30%, and 40%) have been taken for fabrication.

It reveals that, the bulk density of quartz and polyester composites increases from 0.89 gm/cm³ to 1.64 gm/cm³ when the amount of quartz increases from 10 to 40%. This may happen due to possible smallest size of (micron size) in quartz grain. Various researchers have reported that coat the polyester resin with something that does not allow them to agglomerate and repealed each other. The components should be compacted with agitation or pressure, and any air remaining between the particles should be removed by applying vacuum [23].

The effect of immersion time on water absorption of QPCs is shown in Fig 3.

Fig. 3 shows that the water absorption depend on quartz content, immersion of time and amount of materials. It reveals that the water absorption values decreased with an increasing in amount of quartz in the composite. In this view, the rate of water absorption is very low with time. This is because, with an increase in molding load the as prepared composite becomes more dense, i.e. reinforced materials are distributed properly eliminating all voids [24]. However, after a certain

time the rate of water absorption rate decreased sharply due to reduction in the cured polyester and the degree of cross-linking reaction, which diminishes the void spaces [25]. When a composite absorbs a large amount of water, its mechanical characteristics deteriorate. In this case, the examined sample will be more relevant.

Fig.4 illustrates the effect of addition of quartz on flexural strengths of quartz reinforced polyester composite (QPCs). It reveals that the tendency of flexural strength of QPCs decreased with addition of quartz to the polymer matrix.

From Fig.4 it is also evident that, on increasing the volume fraction of quartz the flexural strength of the composites is lower than that of polyester

resin, so these composites are brittle in nature. The decrease in flexural strength of composites with density increases may be due to loss of crystalline properties of the composite materials, as mechanical strength gets lowered with decreasing of crystallinity. Consequently, for unsaturated polyester resin flexural strength obtained was almost similar to the result of the reported values [26].

Fig. 5 shows the E-Modulus of quartz reinforced polyester resin composites by the addition of different wt% of quartz. It is clearly evident from the graph that, elastic modulus increased with the amount of adding quartz respectively. This may be due to high degree of adhesion between the polymer and quartz. The similar result could be obtained by the various literature review [27].

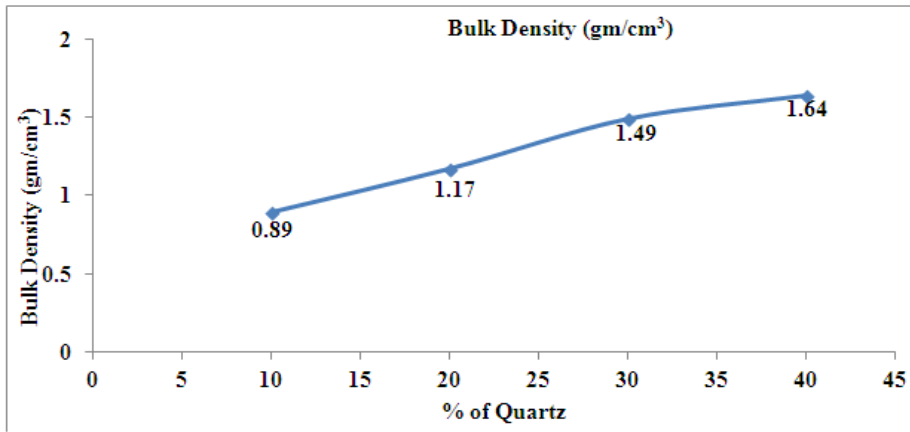


Fig. 2. Bulk density of quartz-polyester composites by addition of different % of quartz

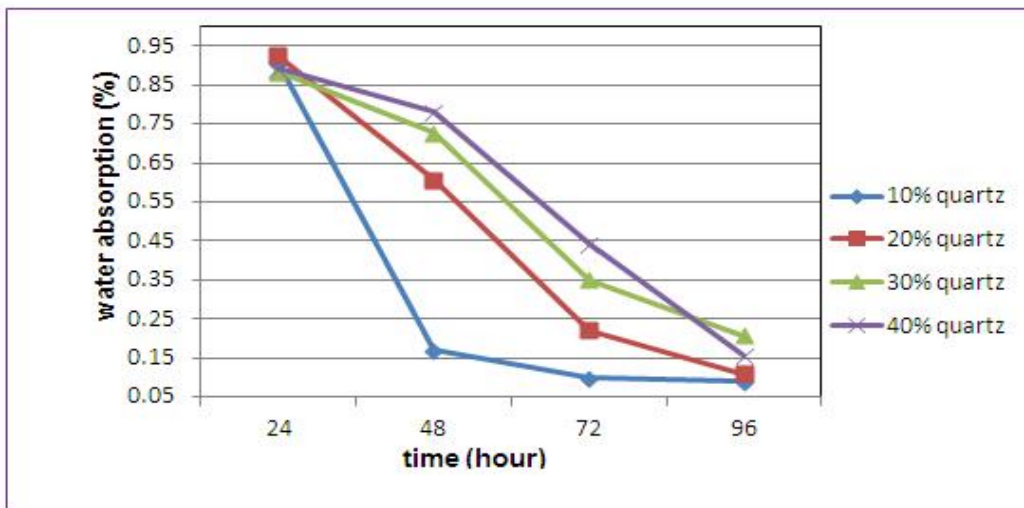


Fig. 3. Effect of immersion time on water absorption of quartz reinforced polyester composite

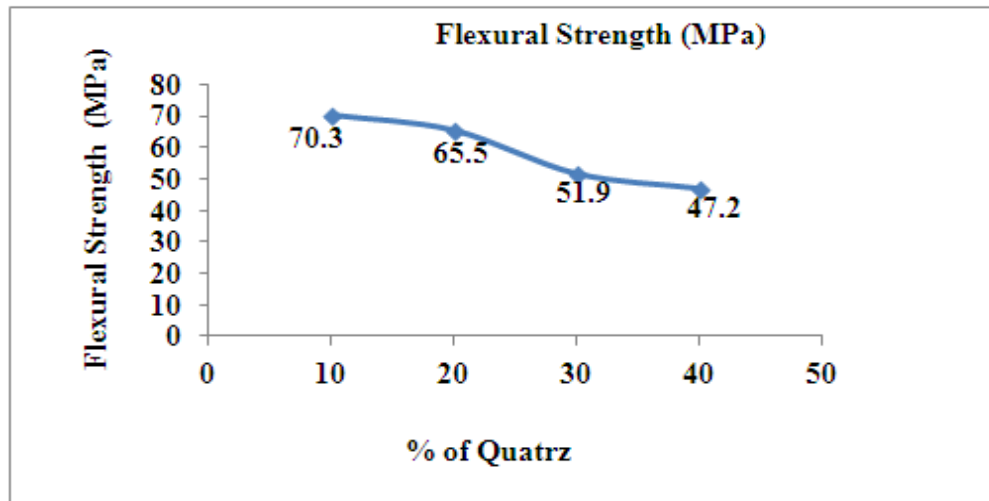


Fig. 4. Flexural strength of quartz-polyester composites by addition of different % of quartz

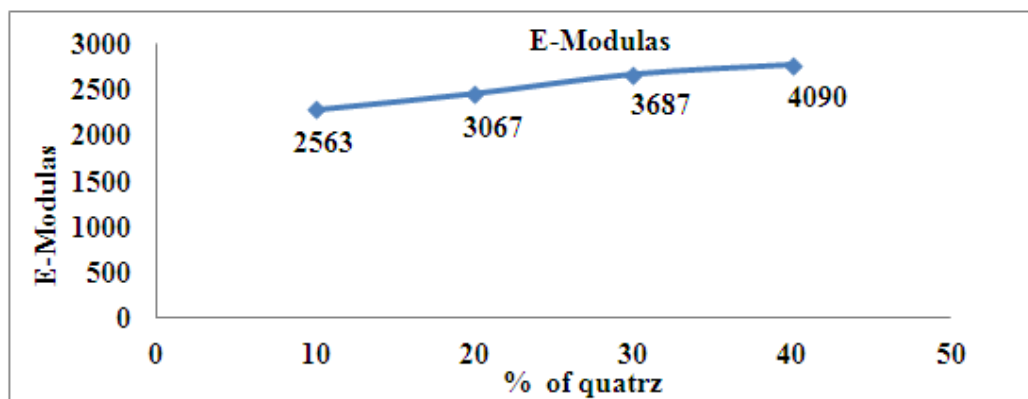


Fig. 5. E-Modulus of quartz-polyester composites by addition of different % of quartz

The effect of addition of quartz on rebound hardness for quartz -polyester composites was illustrated in Fig. 6. Results indicated that Rebound Hardness of QPCs decreased with an increase in amount of quartz added upto 40%. As the stiffness of the quartz filler is higher than that of the polyester polymer, the quartz-polyester resin composites are harder than the pure polyester polymer. According to the above viewpoint, rebound hardness of the composites decreased with an increase in the addition of quartz content due to elastic deformation what is attributed to continuous crystallinity loss [28]. Consequently, the stiffness of the QPCs is high (Higher E-Modulus; Fig. 5) and as a result it is harder.

Fig. 7 illustrates the effect of addition of quartz on tensile strength for quartz -polyester resin

composites. The figure clearly shows that the value decreases with the increase in amount of quartz in the composite. The decrease in tensile strength can be attributed to the physical properties of this filler (quartz) and caused by the aggregation of quartz. Aggregation of quartz is associated to the inhomogeneous distribution of quartz in matrix and reduced the interaction of this filler with the polymer matrix. However, another reason for this is to high viscosity of mixture at high concentration of quartz which caused improper mixing process [29]. In this respect, it indicates that the composite property changes from ductile to tough and brittle due to addition of fillers in polyester resin composite.

Fig. 8 illustrates the effect of addition of quartz on percentage elongation for quartz-polyester composites. The results show that as the

proportion of quartz increases up to 20%, the value falls at first, then increases as the percentage of quartz increases. Exfoliation of quartz from the matrix, which resulted in a porous surface, may have caused the drop in percentage elongation at a concentration of 20

wt%. The reduction in matrix volume is due to the fact that the elastic characteristics are exclusively derived from the matrix [30]. As described in Fig 3, composites with a lower percentage of quartz particle to quartz-polyester were stiffer than those with a greater percentage.

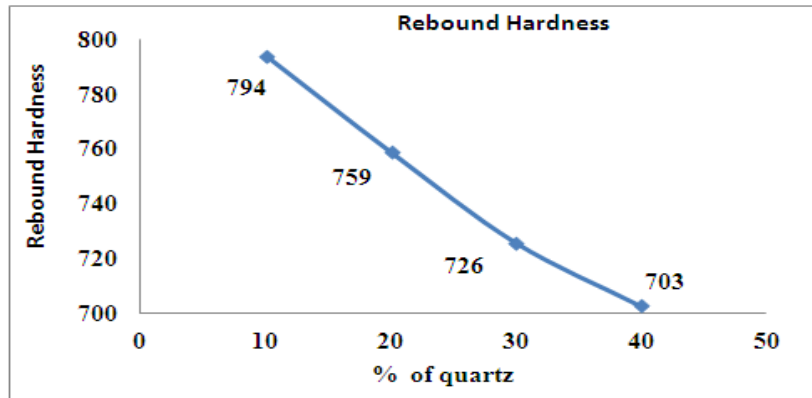


Fig. 6. Rebound Hardness of quartz-polyester composites by addition of different % of quartz

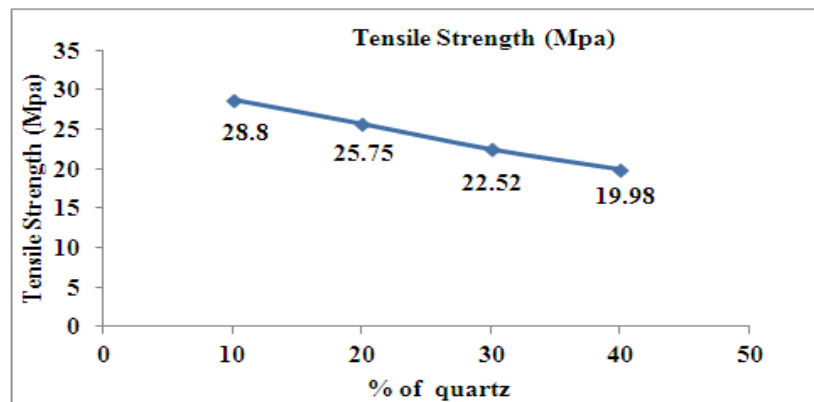


Fig. 7. Tensile strength of quartz-polyester composites by addition of different % of quartz

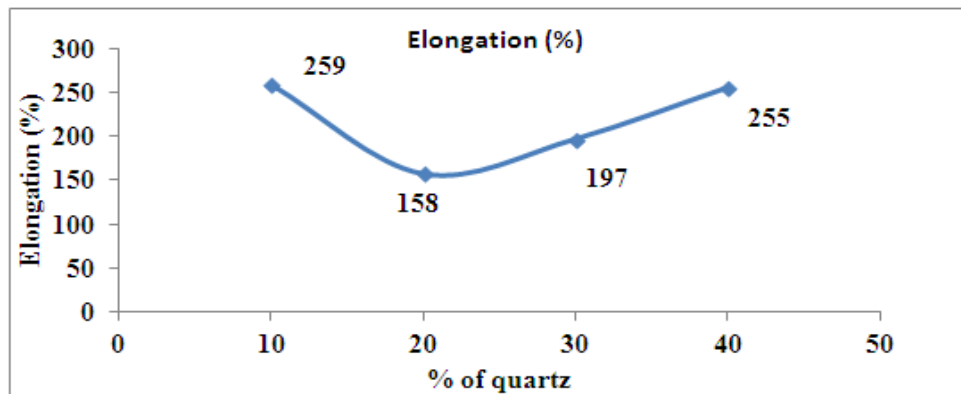


Fig. 8. Elongation (%) of quartz-polyester composites by addition of different % of quartz

The effects of addition of quartz on compressive strengths of QPCs are represented in Fig. 9. Results indicated that compressive strength of quartz-polyester composite at first increases with increasing the percentage of quartz up to 20% then decreases up to 30% and show higher strength at 40% quartz in quartz-polyester composite material. The compressive strength increases due to high degree of adhesion between the polymer molecules and quartz particles. According to literature, the compressive stress rises as quartz content rises due to the catalytic impact of quartz components. While for higher percentage of quartz the compressive strength decreases due to lower magnitude of cross-linking reactions [31].

In overall, from the above results we found that, the water absorption, flexural strength, tensile strength and rebound hardness of the as prepared composite was decreased drastically as the proportion of quartz content in the composite material increased. It has also been observed in previous work by Komimar et al. (1994) that increasing the amount of quartz in quartz filled unsaturated polyester particle composites caused fracture toughness, bending, and compression loading modes to fail, affecting the performance of composite material [32].

The thermo-gravimetric (TG) and derivative thermo-gravimetric (DTG) analyses for polyester resin composites was shown in Fig. 10. The topmost lines represent percent TG, which

indicates the steps of degradation, the middle lines represent DTA, which provides exothermic or endothermic information, and the bottom group of lines represent DTG, which typically provides the maximum degradation rate as well as the temperature for maximum degradation.

The TG graph indicates that all composite samples were deteriorated in a single phase. The 50% degradation temperature of pure Polyester resin was 365.5°C, but that of the QP-1, QP-2, QP-3 and QP-4 were 403.8°C, 402.8°C, 401.3°C and 400.1°C respectively. The sample degraded almost completely between 250°C and 550°C, approximately 83.5 %degradation due to the evaporation of the hydrated water from sample. The main causes of degradation are random scission of cross-linked structures and polymer chain branching [33].

Fig. 10 also reveals that, by increasing the percentage of quartz thermal stability of composites decreases and composite having 10% quartz content shows higher thermal stability than others. The DTA curve of quartz-polyester composites shows one endothermic peak which is due to loss of moisture. The DTG value demonstrated that when the filler content in composite materials increases, the rate of degradation during thermal decomposition decreases. From the DTG data we see that the maximum degradation rate is found for 10% filled composite whereas the degradation rate falls gradually with the increasing of filler content.

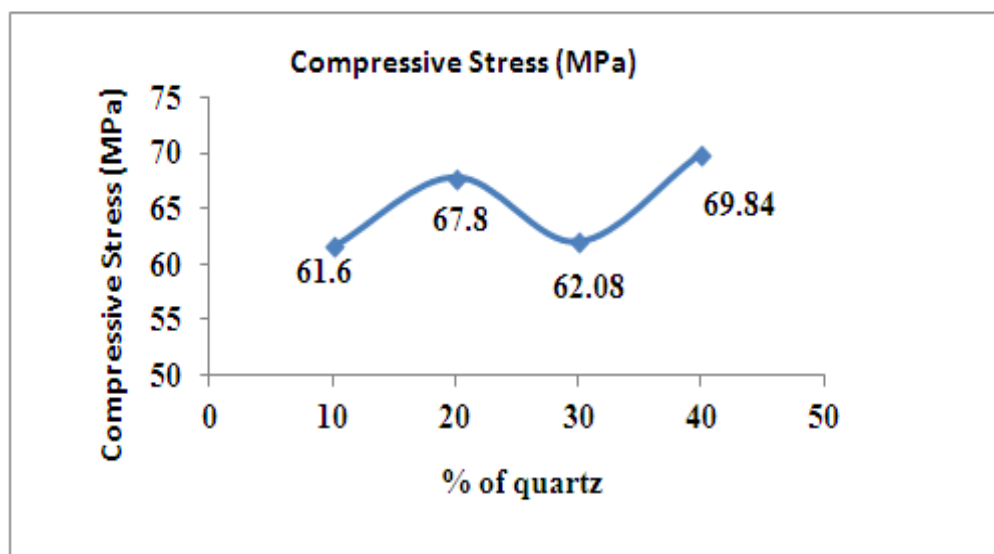


Fig. 9. Effect of addition of quartz on compressive strength of quartz-polyester composite

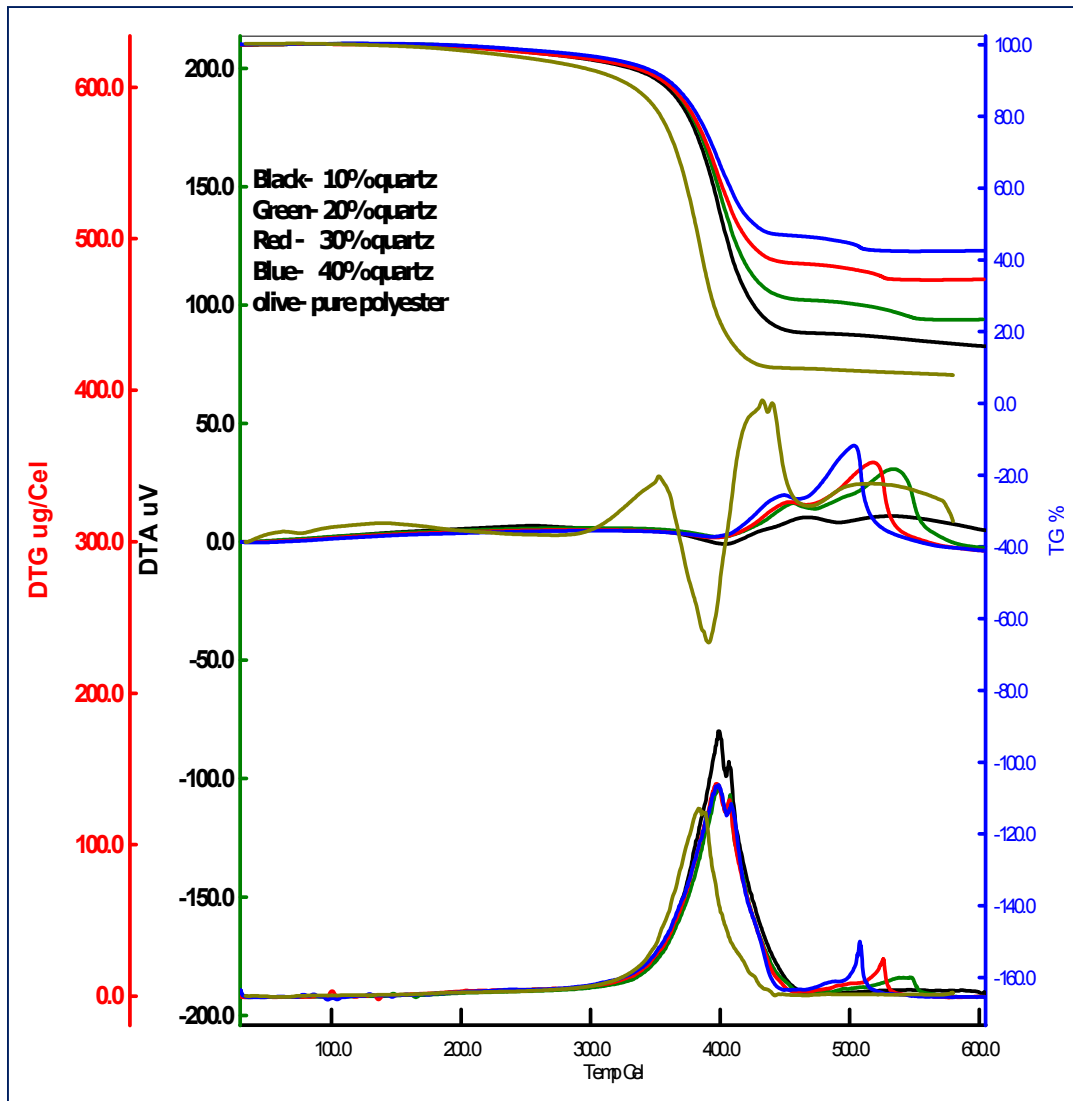


Fig. 10. TG/DTA/DTG for of 10%, 20%, 30% and 40% quartz reinforced polyester composites

Thermo-mechanical analysis of QPCs were carried out and the results are shown in Fig. 11. The topmost lines denotes the TMA line, from which the values of α is obtained used to calculating the slope of the line. The bottom lines represents the DTMA lines, from which the maximum expansion rate and temperature for maximum expansion are calculated. It is shown that the 40% Sample has the highest slope and hence highest α value. The decrease in Tg in Quartz-polyester composites is due to two factors: an incomplete curing process and an increase in the system's free volume. The rise in Tg values due to the inclusion of quartz filler is thought to be due to a decrease in free volume [34].

According to the findings of the experiments, increasing the amount of filler in composites increases the thermal expansion coefficient. The thermal expansion coefficient of a 40 percent volume filled composite was found to be higher both below and above Tg, and therefore across the whole temperature interval of the test. Due to high combustion temperature of quartz, the interaction between the polymer and the filler improves, and this good filler-matrix interaction is responsible for the lower thermal expansion coefficient value, which is also a good indicator of composite stability in a hot environment. Many other have shown similar findings research [35].

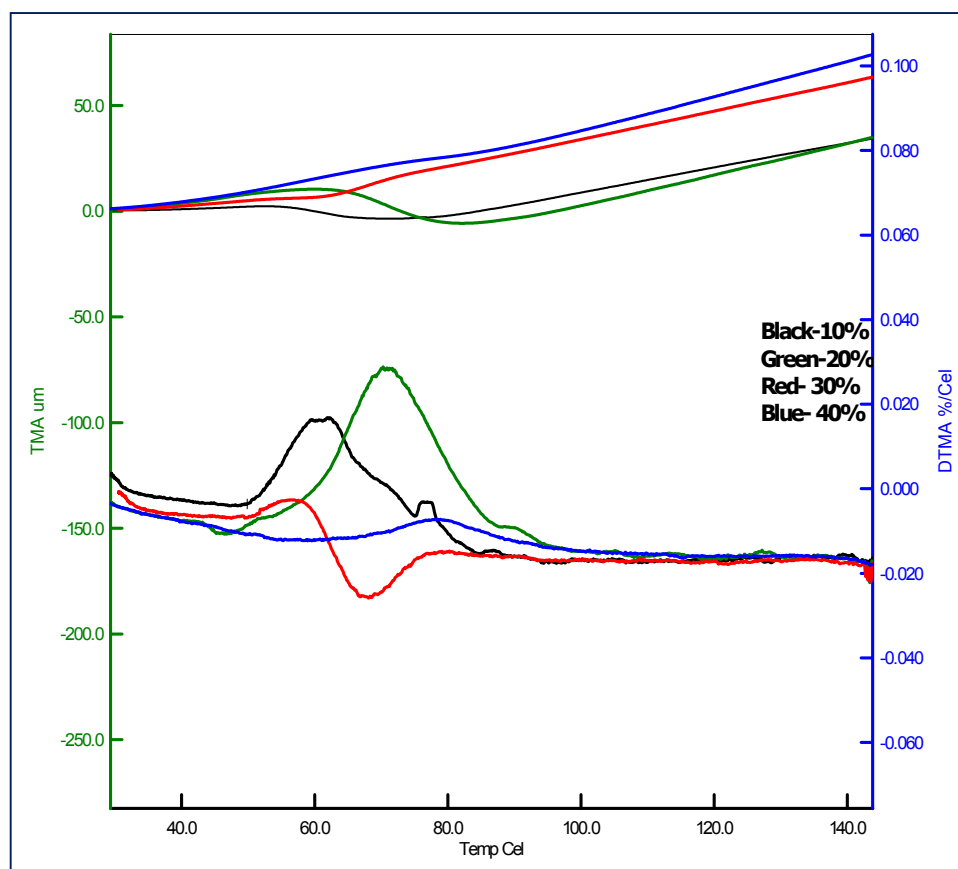


Fig. 11. Comparison of TMA & DTMA curves of 10%, 20%, 30% and 40% quartz reinforced polyester composites

4. CONCLUSION

In this study, quartz reinforced polyester composites were successfully prepared by impregnating quartz with unsaturated polyester resin followed by compression molding process. Significant change occurs in composite physical, mechanical and thermal characteristics while altering the percentage of quartz content in it. The flexural strength and compressive strength of QPCs decreased as quartz content increased. Whereas rebound hardness of the composites decreased due to elastic deformation with an increase in filler content added. Thermal properties of the composite decreased with an increase in quartz content due to the insulating property of quartz. As a result, quartz quantity plays a vital role in evaluating the physico-mechanical and thermal properties of QPCs and a more detailed investigation can be explored to assess their suitability for their applications in various fields.

ETHICAL APPROVAL

As per international standard or university standard written ethical approval has been collected and preserved by the authors.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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