Characterization of Ceramic-reinforced Epoxy-based Composite Materials

[1]A.QUDDOS, [2]A.WADOOD, [2]M.L. MIRZA, [1]M.SHAHID AND [3]S.ZULFIQAR
[1]*Dr.A.Q.Khan Research Laboratories, P.O.Box 502, Rawalpindi, Pakistan*
[2]*Department of Chemistry, Islamia University, Bahawalpur, Pakistan*
[3]*Department of Chemistry, Quaid-e-Azam University, Islamabad, Pakistan*

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Summary: Composite materials based on 2,2-bis (p-2-hydroxy-3-methacryloxpropoxy phenyl) propane (HMAPP) for different applications have been studied. Diametrical tensile strength (DTS), compressive strength (CS) and Vickers micro-hardness (VMH) have been found to decrease with increasing diluent concentration and decreasing filler concentration. Exotherm during setting was found to decrease sharply with increase in filler content for quartz and silica filled composites. Water sorption studies indicate that the amount of water contents are relatively higher for the quartz filler as compared to silica filler. Comparison of mechanical properties show quartz to be better filler than silica for the given formulations studied. The effect of acid and/or heat treatment for quartz filler on the DTS and CS of the final composite have also been studied; the two treatments improved the aesthetic appearance of the composite.

Introduction

Applications of resin-based composite materials in engineering industry are increasing day-by -day by virtue of their superior physical and mechanical properties [1]. The resin plays an important role in determining the thermo-mechanical response of a composite system. Composites are essentially mixtures of filler and a base resin in which the filler may be either in the crystalline phase, vitreous phase or mixture of both [2]. In addition to the resin itself, coupling agents may also be added to coat the filler for better bonding with the base resin. Clinically acceptable handling characteristics can be achieved by diluting HMAPP with diluents (thinners) like triethylene glycol dimethacrylate (TEGDMA).

No single property can give a true measure of the quality of the materials. Several combined properties, determined from the standardized laboratory and service tests, are employed to give a measure of quality and are of great importance in the clinical evaluation of the particular product. However, mechanical properties greatly determine the behaviour of composite materials [3].

The present investigation aims at the study of the mechanical properties such as DTS, CS and VMH of quartz and silica filled HMAPP based composite and the effect of certain formulation variables on them.

Results and Discussion

The variation in DTS, CS and VMH with respect to diluent concentration for quartz and silica filled with composites is shown in Fig.1. An increase in diluent concentration is found to decrease DTS, CS and VMH for both quartz and silica filled composites. During preparation of the formulation, it was observed that concentration below a certain percentage of diluent creates difficulty in making a uniform mixture of the paste. Hence, formulations were prepared using an optimum amount of diluent as a minimum, for making a compromise between ease of mixing and required properties. Keeping in view this problem, the diluent concentration was not decreased below 23% for all the formulations.

The effect of filler content on DTS, CS and VMH for both quartz and silica filled composites are shown in Fig. 2 and the effect on the exotherm during setting is shown in Fig. 3. It is found that an increase in the filler amount results in an increase of DTS, CS and VMH. Although the setting is associated with liberation of heat, however, the exotherm during setting is found to decrease considerably with an increase in filler amount. This decrease is due to reduction in reacting mass as more and more filler is incorporated in the resin matrix. Filler also acts as diluent decreasing the maximum amount of energy that can be liberated per unit volume during the polymerization reaction.

Water sorption studies indicate that the amount of water absorbed is higher for quartz filled composites than the silica filled ones (Fig. 4) Under clinical conditions, water absorption leads to an eventual breakdown of the material at the interface with the recurrent decay. It is caused mainly due to the water dissolved in the polymer matrix. However, water sorption can be reduced by increasing the filler content [5,6]. Silica filled composites exhibit higher exotherm during setting than the quartz filled composites. A comparison of the results obtained

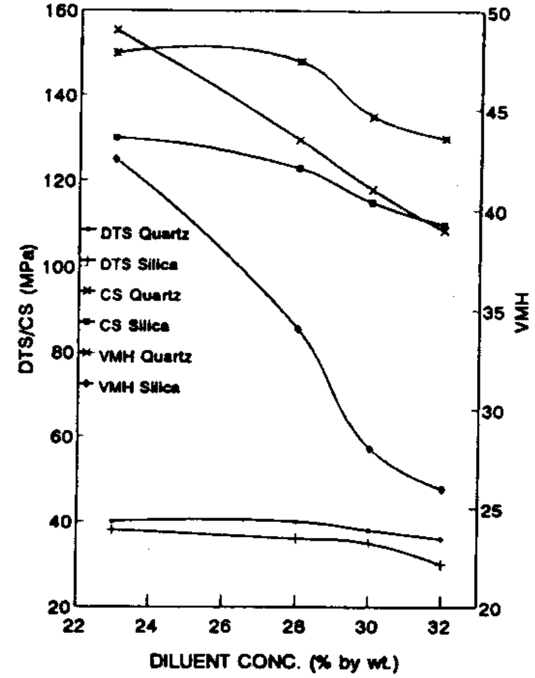

Fig.1

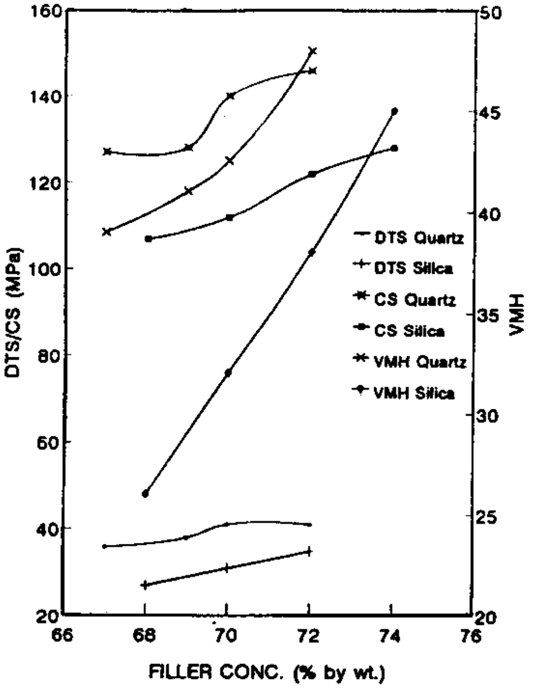

Fig.2

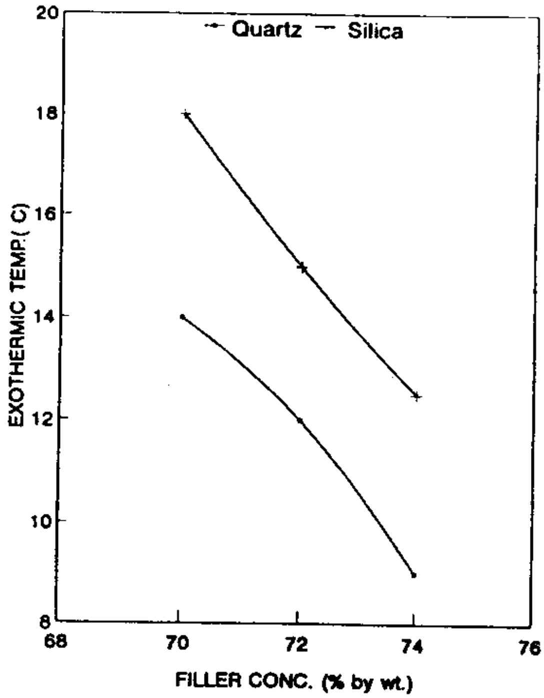

Fig.3

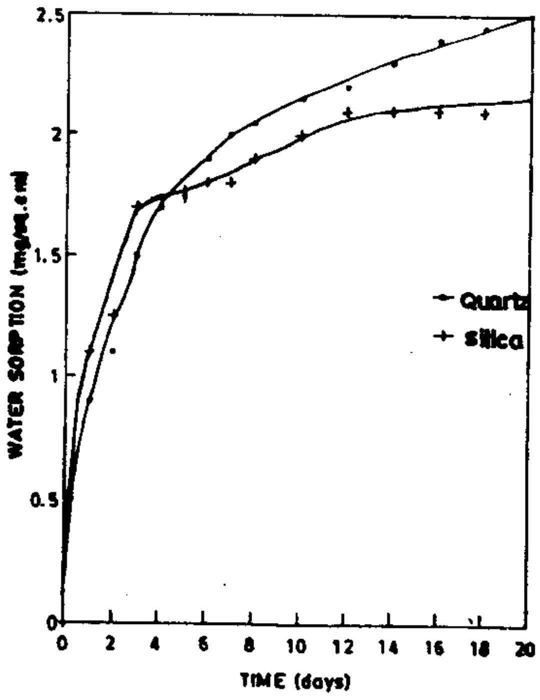

Fig.4

from various properties of all the formulations shows that quartz is better filler in composite materials, than silica.

Filler procured from different sources exhibited difference in properties due to the presence of impurities. Acid treatment burns off the impurities and improves the colour of the filler. The effect of acid and an/or heat treatment of quartz on the DTS and CS of the final composite is shown in Fig.5. Heat treatment of the filler prior to the formulation resulted in an increase in CS of the composite. But a considerable increase is not observed in case of DTS. Acid treatment alone was found to decrease the CS. Besides, both successive treatments could not improve the CS either. However, the combined treatments resulted in aesthetically improved appearance of the samples.

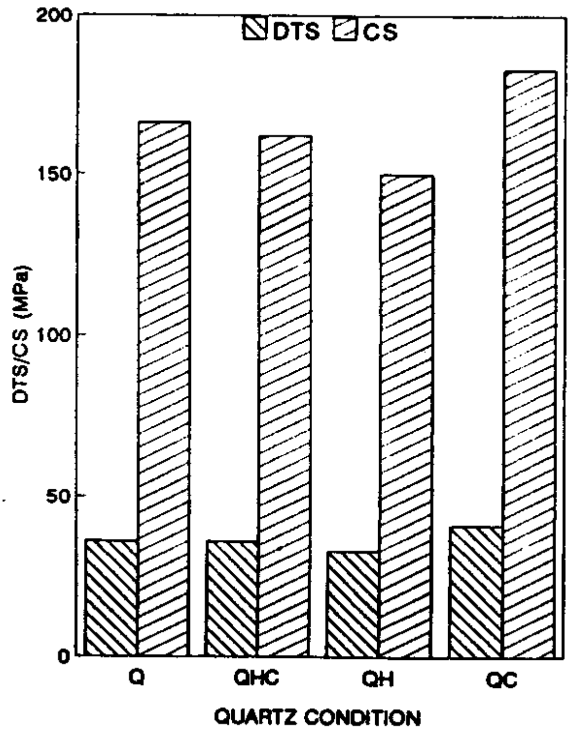

Fig.5

Experimental

Preparation of Composites

Composite resins were formulated in a laboratory at Quaid-e- Azam University, Islamabad, in the form of two-paste systems using HMAPP resin which was synthesized according to the procedure reported elsewhere [4]. Additives like inhibitors and stabilizers were also incorporated in the HMAPP diluted with TEGDMA. Known amounts of inhibitors and activators were added to each half of the above mixture. Equal amount of the fillers with particle size in the range of 20-36 microns, treated with silane coupling agent prior to formulation, were mixed in each of the above halves to get paste A and paste B respectively.

Variation in Formulation

(a) Diluent Variation.

Four formulations were prepared using different concentrations of the diluent(TEGDMA).

(b) Filler Variation.

Four formulations were prepared having different concentrations of quartz and silica (obtained from Amrut Industrial Products).

Determination of Properties for Quartz and Silica Filled Composites.

Mechanical properties (DTS, CS and VMH) were determined for composites containing either quartz or silica as filler. Five samples from each formulation were used to determine water sorption. The exotherm during setting was also noted for quartz and silica filled composites.

Acid and/or Heat Treatment of Quartz Fillers

Four different formulations, prepared using quartz fillers subjected to various treatments, were studied. Quartz used (1) QH (acid treated), (2) QC (heat-treated), (3) QHC (acid and heat-treated) and (4) Q (untreated). Acid treatment was performed by washing the filler with 75% HCl. It was then repeatedly washed with water, dried and then silane treated. Heat treatment was accomplished in a muffle furance at 800°C for 4 hours and then silane treated. DTS and CS were determined for every formulation. Specimens were prepared and DTS and CS determined using a universal testing machine (INSTRON). DTS was then computed using the relationship

$$DTS=2P / \pi DL$$

Where P is the load at fracture, D is the diameter of specimen in mm and L is the length of specimen in mm. A minimum of ten samples was studied for each formulation. Following relationship was used for determination of

$$LCS = load/Area$$

A minimum of ten samples were studied for each formulation. Water sorption (WS) was calculated using the relationship

$$Ws = (Wt - Wo) / A$$

Where Wt is the weight in mg, after immersion, Wo is the conditioned weight in mg and A is the surface area of sample in cm^2. Five discs were examined for each material. Discs were prepared as in the case of water sorption study and the VMH was determined using the MHP- 160 micro-hardness tester, (Carl Zeiss, Jane). The 136 degree diamond pyramid shaped indentor was forced into the material with a load of 100 mg and Vickers Hardness value calculated using the relationship

$$VMH = 1854.4\ P / d^2$$

Where P is load in grams and d is diagonal of the indent in microns. VMH values were determined for formulations using four different filler concentrations for quartz and silica filled composites. Pastes A and B were mixed and kept in Teflon mold. Peak exotherm was recorded during setting. Using a 24- gauge Copper-Constantan thermocouple.

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