Progress in Natural Science: Materials International xxx (xxxx) xxx–xxx

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Contents lists available at ScienceDirect

Progress in Natural Science: Materials International

journal homepage: www.elsevier.com/locate/pnsmi

Investigation of functional, physical, mechanical and thermal properties of TiO_2 embedded polyester hybrid composites: A design of experiment (DoE) study☆

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ARTICLE INFO

Keywords:
Hybrid composites
Polyester
Design of experiment
Titania

ABSTRACT

This paper presents a study on the Design of Experiments (DoE) approach to optimize the fabrication parameters of titania (TiO_2) embedded glass fiber reinforced polyester hybrid composites (HCs). HCs of unsaturated polyester resin (UPR) were fabricated using methyl ethyl ketone peroxide (MEKP) as the curing agent by hand lay-up process (HLUP) and compression molding technique (CMT). The fabrication parameters were optimized by Taguchi DoE (an orthogonal array of L_{25}) using 3 control factors (concentration of TiO_2, concentration of MEKP and curing temperature) having 5 levels each. Statistical tools were employed to identify significant factors affecting tensile strengths and its reproducibility during HLUP. It was found that the concentration of TiO_2 in HCs significantly influenced the tensile strength (TS) followed by MEKP concentration and curing temperature. The highest value of TS was obtained at 3 wt% TiO_2, 2 wt% MEKP and 80 °C. Nevertheless, the sensitivity of TiO_2 concentration was basis for fabrication of polyester titania glass fiber hybrids (PTGFHs), which were further investigated for density and void fractions, linear shrinkage, flexural strength, impact strength, hardness and thermal behaviors. Moreover, cross-linking and hydrogen-bonding between polymeric chains, styrene, silica content of glass fiber and TiO_2 particles in PTGFHs were confirmed by Fourier transform infrared spectroscopy.

1. Introduction

The association of inorganic fillers in the glass fiber reinforced polyester composites (GFRPCs) improve the physical mechanical and thermal characteristics [1]. Several metal oxides are discussed in literature such as zirconium oxide, zinc oxide, aluminum oxide, silicon oxide and cerium oxide [2]. However in recent era, titanium oxide/ titania (TiO_2) has attracted as inorganic phase for the fabrication of titania embedded glass fiber reinforced polyester hybrid composites (HCs), due to its specific properties such as photocatalytic, high refractive index and UV radiation absorption [3]. The properties of glass fiber like high electrical and corrosion resistance with good dimensional stability are responsible for its use as reinforcement in GFRPCs [4]. The complexities in the design and manufacture of HCs have limited their widespread applications [5]. For example, improper loading of filler and curing agent in matrices, insufficient adhesion between polymer, fiber and filler, void formation, poor wettability and improper processing [6]. Fu et.al [7] observed the mechanical and thermal properties depended on the loading of filler and the amount of curing agent. Huang et.al [8] revealed that the curing temperature was also an important processing factor that significantly influenced the characteristics of GFRPCs. Therefore, optimum synthesis and processing parameters must carefully be selected in order to fabricate the perfect composite products [9]. The conventional approach to experimental work is to change one factor at a time, keeping all other factors constant, this system does not yield satisfactory outcomes in a wide range of experimental settings as well as material and time consuming [10]. Therefore, Taguchi Design of Experiments (DoE) approach is

☆ Peer review under responsibility of Chinese Materials Research Society.

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https://doi.org/10.1016/j.pnsc.2017.12.005
Received 10 August 2017; Received in revised form 28 December 2017; Accepted 29 December 2017

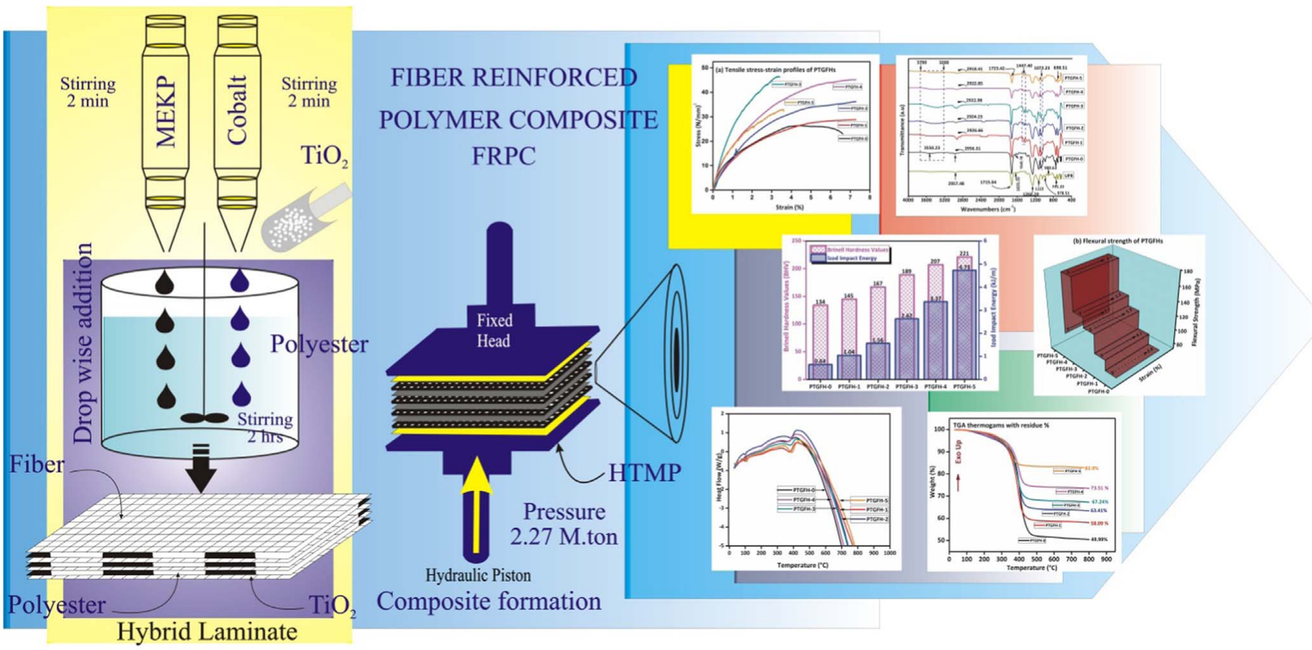

Fig. 1. Schematic diagram of experimental set-up for fabrication of composites using hand lay-up process (HLUP) and compression molding technique (CMT).

introduced so that, the synthesis and processing parameters are optimized in time efficient manner. Moreover, obtained data can be analysed to yield deeper insight of the processing parameters and their effect on the desired set of properties [11].

The main focus of this study is to fabricate TiO_2 embedded glass fiber reinforced polyester hybrid composites (HCs) by utilizing DoE approach; preliminarily on tensile testing for construction applications. In DoE approach for tensile testing, we investigated the effect of the three control parameters having five levels each: titania concentration (1 wt%, 2 wt%, 3 wt%, 4 wt% and 5 wt%), MEKP concentration (0.5 wt %, 1 wt%, 1.5 wt%, 2 wt% and 2.5 wt%) and curing temperature (60 °C, 65 °C, 70 °C, 75 °C and 80 °C). These parameters were determined by using cause and effect diagram (data not shown here). It was observed that A3 (3 wt% of $TiO_{2)}$, B4 (2 wt% of MEKP), and C5 (80 °C) had the 'best' result based on the tensile properties. Furthermore, TiO_2 concentration was the most striking parameter on the basis of maximum-minimum response found for the signal to noise ratio (S/N) of the tensile strength. HCs of polyester titania glass fiber hybrids (PTGFHs) were fabricated by varying TiO_2 concentration (1–5 wt%), with 2 wt% of MEKP and 80 °C curing temperature fixed as shown in Table 5. All HCs were fabricated by hand lay-up process (HLUP), combined with compression molding technique (CMT) at prescribed parameters of DoE approach. Reproducibility and statistical confidence of the HCs produced from Taguchi array (L_{25}) were determined by the mean response for standard deviations at each run. This research considered the both external factors (controllable factors such as matrix concentration, fiber concentration, fiber orientation and filler concentration) and internal factors (operating temperature, humidity, human error). It can be firmly believed that detailed analysis of the internal factors affecting synthesis process, which may have very small effect but their appropriate control will offer the process reproducibility and integrity of the laminates to higher extent.

2. Experimental program

2.1. Materials and specimens preparation

The unsaturated polyester resin (UPR) (pre-polymer of maleic anhydride and propandiol) with average molecular weight (Mw = 1500 g/mol) having of 40 ± 2% styrene content and inhibitor hydroquinone (150–200 ppm) was obtained from Aropol, Ashland Chemical Hispania, Spain. Cobalt naphthenate (CN) as accelerator and methyl ethyl ketone peroxide (MEKP) as catalyst (curing agent) was acquired from Fluka Co. Glass fiber (E-glass) roving strand mat was acquired from Toray, Japan. Micro-TiO_2 particles (melting point ~ 1843 °C and 4.23 g/cm^3 density) were purchased from Sigma Aldrich.

The TiO_2 in predetermined weight concentrations (0 wt%, 1 wt%, 2 wt%, 3 wt%, 4 wt% and 5 wt%) and accelerator (1.5 wt%) were dispersed in the UPR by a high-speed mixer, run with 2000 rpm at 50 °C for 2 h. The dispersions were cooled to room temperature following by the addition of MEKP (0.5 wt%, 1 wt%, 1.5 wt%, 2 wt% and 2.5 wt%) respectively and then mechanical stirring for 2 min for uniform mixing of curing agent.

Unidirectional (1D) hybrid composites (HCs) laminates were fabricated by a layer-by-layer impregnation using hand lay-up process (HLUP). Four individual sheets of cross plied woven roving E-glass fiber mat with dimensions (180 × 180 mm^2) were immersed in dispersions for complete impregnation of TiO_2 embedded UPR resin including CN & MEKP and then stacked on one another by HLUP in compressed die of stainless steel (SS). The whole assembly of HCs laminates (along SS die) was kept in a high temperature melt press (HTMP) under 5000 pounds (2.27 metric tonne) at 60, 65, 70, 75 and 80 °C respectively for 2 h to get complete curing and uniform thickness. Preparation of HCs laminates was tricky practice so a plain geometry of SS was implemented for sampling. After molding in HTMP, the whole assembly of HCs (along SS die) was cooled to room temperature at same pressure for avoiding thermal residual stresses. And all HCs were prepared with same number of fiber sheets. The schematic diagram of experimental set-up is shown in Fig. 1.

2.2. Test apparatus for tensile testing

Universal Testing Machine (UTM), Testometric Model FS100 CT UK with 100 kN load cell was used to evaluate the tensile properties under a static load according to ASTM: D 3518-0. The UTM was operated at a speed of 2.0 mm/min at ambient temperature conditions during tensile testing. Three trials were conducted for each specimen individually in the UTM and the average values were used.