



EFFECTS OF UV CURING EXPOSURE TIME TO THE MECHANICAL AND PHYSICAL PROPERTIES OF THE EPOXY AND VINYL ESTER FIBER GLASS LAMINATES COMPOSITES

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ABSTRACT

Ultra-violet (UV) curing process is introduced in the curing of polymer matrix composites (PMC) for the application in producing bullet proof vest. Two types of adhesives or matrix materials were used: epoxy and vinyl ester. Each of them were mixed together with different types of photoinitiator; Bisacyl Phosphine Oxide (BaPO) and Alpha Hydroxyl Ketone Peroxide (AHK) at 1.0 per hundred of resin (phr) from the total proportion of the mixture. Sandwich construction of the PMC was done by hand lay-up process where the mixture was wiped on the fiber layers. Samples were then cured under various duration times which are 3, 6, 9 and 12 minutes to compare the overall quality of produced composites under the exposure of the UV light. The laminate was then tested to determine its characteristics of physical properties and its behavior to the applied loads. Morphological observation through Scanning Electron Microscope (SEM) was performed in order to evaluate the quality of adhesion between each fiber layer and matrix wetting behavior. Vinyl ester is not recommended to be used as the matrix since two days is required to make it fully cured unlike epoxy which was cured rapidly once exposed under UV light. On the other hand, the effect of curing time shows the optimum result for density was obtained at 12 minutes of curing time. Curing time between 6 to 9 minutes is found to be optimum for tensile strength value. Three minutes of curing under UV light exposure caused maximum value in penetration resistance or hardness for the resulted composite laminates.

Keywords: UV curing, epoxy, vinyl ester, photoinitiator, curing time effects, mechanical, physical, morphological.

INTRODUCTION

Ultraviolet (UV) curing is known to be a hardening process of liquid material when it is exposed to UV radiation. The curing process of polymeric material by using UV light has played an important role in the processing of polymer material since the process is more advantageous than heat cure. Although curing by this technique is relatively slow compared to heat curing, the result is high strength and high impact properties of Polymer Matrix Composites (PMC) due to crystallinity enhancement of the polymers through cross-linking mechanism. One of the applications of the curing process of polymeric material by ultraviolet light can be seen in producing personal protective equipment (PPE) for instance bullet proof vest. This research is conducted as the preliminary stage in producing the bullet proof vest where the PMC will be used to wrap an alumina plate in order to act as holder when high ballistic impact hit the bullet proof vest. This will avoid alumina plate break into pieces once bullet is triggered to hit them.

The objective of this research is to develop an outside part of bullet proof vest and to assess the ability of the UV light in converting the thermosetting material to become harden or cure under the UV exposure with the assistance of photoinitiator. In addition, it is also to study the parameter of curing time in the UV curing process of polymeric materials and its effects to the properties of the resulted products. This study has its own importance and benefits, which are to provide the fundamental

understanding on the effects of UV curing to the physical and mechanical properties for the application of bullet proof vest manufacturing technique. The output of the cured PMC will then be studied and analyzed.

Although a particular substance to be processed may vary widely depending upon its application and the final use, they are basically composed of polymer. UV curing, a conversion process of polymeric materials from a liquid to solid by UV light is a popular alternative instead of conventional drying. The number and variety of applications for UV curable inks, coatings, and adhesives, continue to expand at a rapid pace and pose new design challenges to increase cure efficiency, speed, and the physical properties of the cured polymer film. UV curing is highly adaptable to painting and coating, decorating, and assembling of a great variety of products owing to some of its key attributes. It is a low temperature process where heat is not required and a high speed process cure is nearly instantaneous. In addition, it is energy efficient processes whereby energy is invested only in the curing reaction, not in heating [1]. UV curing adhesive also have two components. One part is the adhesive resin itself and the second part already mixed in is called a photoinitiator. The secret of the photoinitiator is that, it will not react with the resin by itself. The photoinitiator must absorb UV light before anything can happen. When the UV light is delivered, the photoinitiator will undergo a chemical reaction and produce products that cause the adhesive to harden better. One type of photoinitiator used in this study



is BAPO with Irgacure 819 as its trade name. BAPO is a versatile photoinitiator for radical photo polymerization of unsaturated resins upon exposure to UV light. It has demonstrated useful application in white pigmented formulations, the curing of glass fiber reinforced polyester/styrene systems and for clear coats for outdoor use with light stabilizers. The outstanding absorption properties of BAPO also allow curing of thick sections [2]. On the other hands, AHK is the second photoinitiator that used in this research with trade name Irgacure 184. This is highly efficient non yellowing photoinitiator, which is used to initiate the photopolymerisation of chemically unsaturated prepolymers in combination with mono or multifunctional vinyl monomers [2].

UV light has two important characteristics which are wavelength and intensity. For the photoinitiator to react correctly, it must be exposed to light of the correct wavelength and of sufficient intensity. Otherwise, the chemical reaction may not happen completely. The result will be poor or inconsistent in the adhesive performance [3]. UV curing is dependent on photon molecule collision. The successful implementation of any UV curing process is dependent on the ease or difficulty of projecting photons into a curable material [1]. The optical properties of the curable material (polymeric resin) and the optical characteristics of the lamp must be matched to produce an effective UV curing system. The UV energy striking the surface causes the photoinitiator to trigger the polymerization reaction. The material is usually solidified or dried when it exits the UV cure zone. The time, and consequently the space, required for cure is significantly less than thermal drying methods. Because the process relies on UV light to initiate the crosslinking of molecules, it does not evaporate any solvents nor significantly heat the substrate [4]. The application, particularly the end product it produces, will determine the requirements of the physical properties of the cured photochemistry [2]. UV processing offers several advantages over other finishing methods. Typical product lines involve coatings (on wood, metal, paper and plastic), inks (for letterpress, lithographic, gravure and screen printing) and adhesives (for film, foil or paper substrates). The industries using

these technologies are diverse and varied, including automotive components, medical products, electronics, CDs and DVDs, two piece and three piece can print, pipe and tube coating, furniture, fiber optics, flooring, packaging and containers [4].

MATERIALS AND METHODS

Raw materials and sample preparation

Polymeric resins are the main raw material that was mixed together with photoinitiator. Both epoxy and vinyl ester was act as binder in the mixture. It has been bought from Wee Tee Tong Chemicals Pte. Ltd, Singapore. Meanwhile, photoinitiator material has been sponsored by Ciba Specialty Chemicals, Singapore. The photoinitiator was added to the matrix materials in the amount of part per hundred of resin (phr), with ratio of 1:3. There are two types of samples that involved in this research. Sample B is a series of sample with vinyl ester as the matrix material while sample C is the sample with epoxy as the matrix material. Details on sample formulation are described in the following Table-1. Each sample B and C was mixed together and stirred to obtain homogenous solution. The total weight of resin is 120 gram. The composition ratio of BAPO to AHK is 1:3 and added in the amount of 1.0 phr from the total mixture. At the first stage of sweeping process, a layer of woven roving was placed on a flat surface. Once mixture of resin and photoinitiator is homogenized, it was swept over the top of the glass using brush. After that, another layer was put on the first glass laminate and the same procedure was repeated until the laminate of 10 layers is produced. This laminates construction was done repeatedly for another sample. After the 10 layers of woven roving stick together, the laminate was then undergoing vacuum bagging process where it was put in a vacuum chamber. By performing this step, the adhesion between the layers was improved and the entrapped air was expelled. After the vacuum bagging stage, the glass laminate was exposed to UV light. Once the process completed, sample was taken out and ready for testing.

Table-1. Details on sample formulation and curing strategy.

Sample	Details for each sample
B	Woven roving + Vinyl ester + BAPO + AHK ; (BAPO + AHK = 1.0 phr) 3 minutes 6 minutes 9 minutes 12 minutes
C	Woven roving + Epoxy + BAPO + AHK ; (BAPO + AHK = 1.0 phr) 3 minutes 6 minutes 9 minutes 12 minutes



Sample testing

Density test

Density test was carried out in order to study the physical properties of UV cured laminate composites produced with parameter that has been varied for instance type of adhesive used to develop sandwich construction between each layer of glass as well as the time of exposure under UV light. This test was conducted by applying the Archimedes principle.

Tensile test

Tensile test was carried out in accordance to ASTM D 3039 standards. The test was carried out at a crosshead speed of 2 mm per minute. Specimens for the tensile test were produced by cutting out laminate strips measuring 200 mm x 25 mm. The specimens were cut 3 mm oversize and final dimensions obtained by grinding and using sand paper to produce smoother end surface. Aluminum end tabs of 3.2 mm thick and measuring 50 mm x 25 mm was locally bonded onto the both ends of each laminate. A total of 8 specimen strips were cut from each of the composite laminates produced. For this testing, two series of sample were produced. Series B produced with vinyl ester as the matrix material while series C has epoxy as the matrix material. The purpose of this testing is to study the effects of resin used and to study the effects in curing by increasing the exposure time under UV light. Figure-1 below shows samples for tensile testing.



Figure-1. Specimen for tensile test ASTM D 3039.

Hardness test

Hardness test was performed to the samples in order to know the resistance of surface penetration to the glass laminates that has been cured under UV light. The hardness test was conducted in the Brinell hardness test mode with diameter of indenter is 10 mm and 100 N of loads.

Morphological observation by Scanning Electron Microscope (SEM)

The purpose of this observation is to determine if proper adhesion and fiber wet out occurred in each of the laminates. SEM used for this observation is the variable pressure scanning electron microscope.

RESULTS AND DISCUSSIONS

Physical testing of density behavior

The purpose of the density test is to observe the effect on the physical properties of UV cured laminate composites produced by different type of resin and to study if variability exists in the variation of curing time under the UV light. Generally, the density of the vinyl ester used is 1.35 g/cm^3 while for the epoxy; the density is 1.50 g/cm^3 . The samples using epoxy as an adhesive have greater density as compared to samples using vinyl ester. This result is in accordance with the density data of both virgin resins where epoxy's density has greater value than vinyl ester. Figure-2 shows the density behavior of PMC in function of curing time under the UV light to produce composites of samples B and C. Based on the plot, the density value for sample B and C seems to increase constantly with the increasing of curing duration. For both sample B and C, the plotted data shows an increment as a time of curing increased. This indicates that the exposure time under the UV light affects the density value of UV cured laminates. It may caused by the atom of resin that might have enough energy to move, in order to make better arrangement between them while curing is in progress. In addition, this will allow the fibers to make better linkage due to curing with polymer chain as time of UV light exposure increased. Chain in the resin will have more time to arrange the fiber closer to each other. Thus, the probability for the void to exist was minimized by longer exposure time of UV light. The void occurred preferentially at the ply interfaces and grew quite large [5].

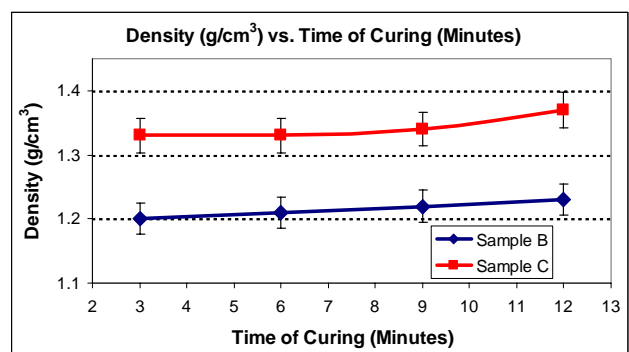


Figure-2. Density of samples at various curing time exposure.

Mechanical testing of tensile strength

Figure-3 shows the plotted data for tensile strength versus time of curing for both samples in group B and C. From the graph, both of the tensile strength curves showed the strength peaks at certain value of exposure



time. Therefore, in overall, distribution data shows that, series of sample C have greater tensile strength compared to the series of sample B. From the plotted graph, the value of tensile strength for 3 minutes of curing exposure is the lowest compared to all data obtained in the testing result for the series of sample B. This might be due to not enough curing time of 3 minutes for the adhesive to complete the transformation phase from liquid to solid under the UV light exposure. Thus, it produces weak bonding between each fiber between the polymer matrixes. The glass fiber woven roving inside the laminate is still wet, thus not strongly bonded between each other. For 6 minutes of exposure time, the mechanical tensile strength of composites for both polymer matrixes were getting stronger compared to 3 minutes of curing exposure. The wet fibers become completely dried, making the adhesion bonded strongly. For further 3 minutes of curing exposure, for the sample B3, the tensile strength was found to increase further, however for curing time up to 12 minutes, the tensile strength decreased. At 12 minutes of curing time the fibers become charred where it will not act as reinforcement and the ability to bond between each particle become limited.

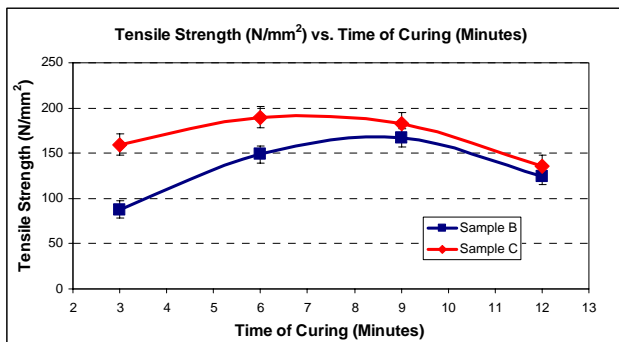


Figure-3. Tensile strength of samples at different curing time exposure.

For sample C1, 3 minutes of curing under the UV light exposure shows greater tensile strength. This is due to the type of adhesive where epoxy has better properties compared to vinyl ester. The adhesive between the fibers is strongly bonded for epoxy resin. However, the limitation of the epoxy is that, it will not cure as rapid as vinyl ester. After UV curing step, it needs another 2 days of drying period at room temperature to be fully cured and hardened. After another 3 minutes of curing exposure, the tensile strength was increased further. This means that, higher force is needed to break the sample because better properties were achieved for sample C2. As the exposure time increased, the result showed a constant value of tensile strength but there was a bit reduction occurred at 9 minutes of exposure time. This means that for epoxy resin, the sample achieves peak value between 6 to 9 minutes of exposure time. As the exposure time increased until 12 minutes, the value of tensile strength was decreased.

Hardness test evaluation

Figure-4 shows the plotted data for hardness of produced composites that was cured under the UV light with different exposure time. Series of sample B represent samples that were wiped with vinyl ester as adhesive resin and series of sample C represent samples that were wiped by epoxy as adhesive material. Based on Figure-4, the plotted graph shows that epoxy which is represented as series of sample B have greater hardness value. For series of vinyl ester sample B1 shows that, with 3 minutes of exposure time, the hardness is better compared to sample B2 even B2 was exposed under the UV light for longer period which is 6 minutes. The comparison can be clarified by the idea of uneven adhesive distribution on glass laminate during the wiping process. The resin of adhesive was probably wiped on the glass layers in an uneven condition where the resin distribution may slightly varied at different places on the same plate.

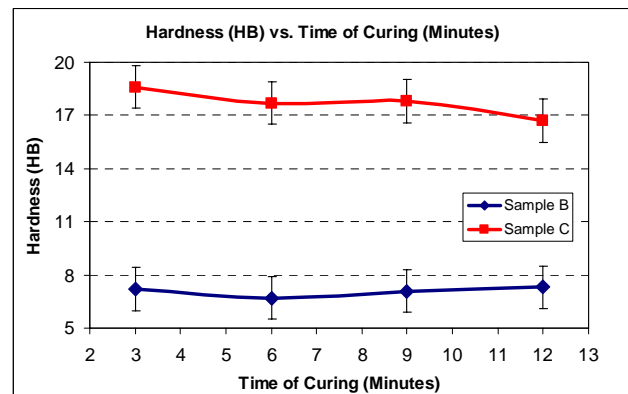


Figure-4. Hardness of samples at different curing time.

In addition, void may exist in the fiber layers that resulted in lower hardness value. Thus, during the hardness test, the result obtained shows that the variation in hardness is depends on the resin distribution on glass laminate. Thicker adhesive wiped on glass laminate may have greater hardness value. For sample B3, which is 9 minutes of exposure time under UV light, there is a bit increment in hardness values as compared to 6 minutes of curing time. Hardness value was increased further when cured until 12 minutes of UV exposure for sample B4. On the other hand, sample C1 has highest value of hardness among other sample in series C. However, for 6 minutes of curing time, the value of hardness for sample C2 shows a bit decrement. The value increases slightly at 9 minutes of curing time for sample C3 but decreases again for sample C4 down to 16.7 HB. Thus, from the data obtained, it is indicated that, with the increasing of curing time, only series of sample that used vinyl ester as adhesive shows an increment in term of hardness. In contrast, the value of hardness for epoxy is decreased. Thus, in overall, it can be clarified that the variation of curing time gives no effect to the hardness of UV cured laminate.



Morphology analysis

In addition to the physical and mechanical properties, morphological observation through the Scanning Electron Microscope (SEM) was also carried out on both of UV cured laminates that use vinyl ester and epoxy as adhesive material. The micrographs of the selected UV cured laminate are shown in Figure-5 and it was taken at the point of tensile fracture. It clearly shows that the fiber strands have very little resin impregnated along their length. This could probably be indicative of improper adhesion between the fiber and the matrix during

the curing time. The surface of fiber is covered by the matrix in an uneven condition. In addition, the existing of void can also be seen. The presence of voids affects the result especially to the mechanical properties of the UV cured laminates. However, at the point of fracture the fiber strands appear to pull out cleanly with little evidence of resin impregnated along their length. No fiber wet out found in the fracture surface of the sample when observation was conducted. All samples have good well dried adhesives, indicates that the matrix solution was cured enough before testing is conducted.

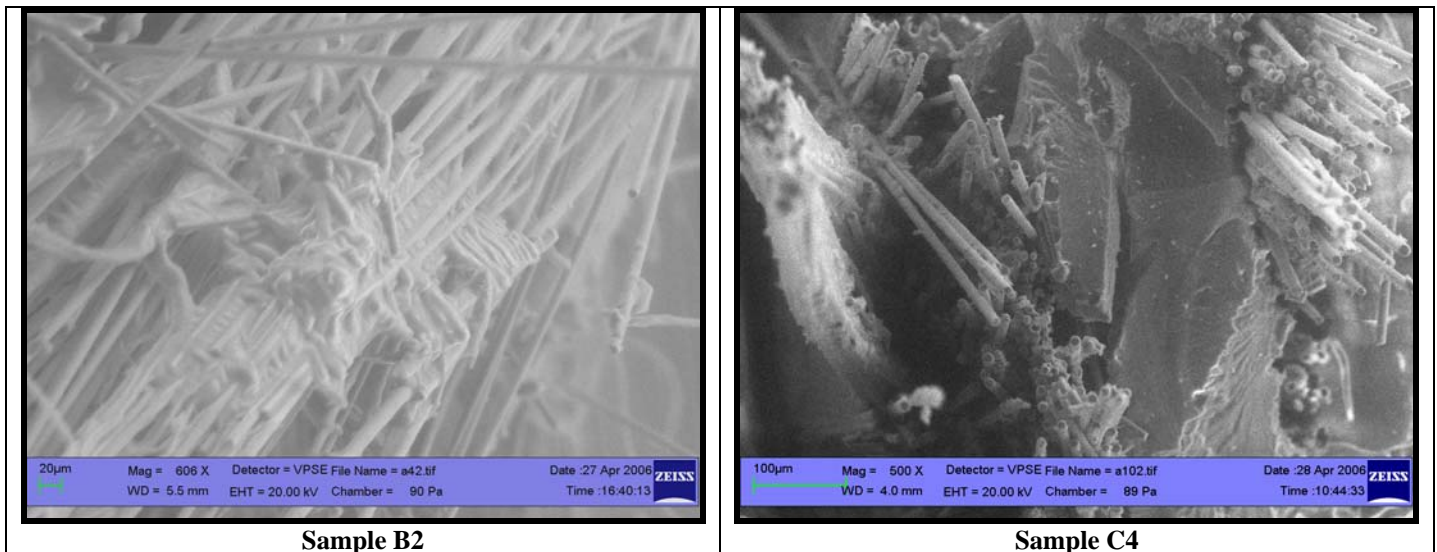


Figure-5. Micrograph of selected sample of UV cured laminates composites.

CONCLUSIONS

In this research, several observations like mechanical and physical properties have been done to the UV cured laminates composites with respect to the processing parameter of UV curing exposure time. It found that the vinyl ester was not suitable to be used as adhesive since it needs two days to be cured after being exposed under the UV light exposure. On the other hands, epoxy can cured rapidly after three minutes of exposure time under the UV light. Thus, the use of epoxy in the application of UV cured laminate composite is a right option if compared to vinyl ester. Moreover, it was clearly showed that the density of epoxy was higher compared to vinyl ester, hence gives greater density of PMC with epoxy as matrix materials. In conjunction with the density properties, as the exposure time under the UV light was increased, the density of the PMC was increased. This tells that, voids within the substrate were reduced or eliminated due to curing mechanism. For tensile strength, the value was higher for epoxy in comparison to vinyl ester. In addition, the tensile strength was reached it peaks value between 6 to 12 minutes of curing time. The curing time to the substrate were found not affects the value of hardness. Furthermore, through the morphological observation by SEM, it was found that proper adhesion is not achieved between the layers of fiber and the matrix adhesive. Overall, further study need to be carried out in order to

fully discover the potential of proposed materials and curing technique especially for bullet proof vest application. Results and properties obtained from this preliminary investigation is still need to be expanded by some other study to achieve the optimum information with regards to the contribution of UV light in converting the matrix materials from liquid to solid.

ACKNOWLEDGEMENTS

The authors acknowledge with thanks the support of CIBA Chemicals Specialty and UTeM for laboratory, raw materials, sample preparation and the technical advices. Special thanks to Universiti Malaysia Pahang for sponsoring this research initiative.

REFERENCES

- [1] Stowe R. W. April 2002. Key Factors in the UV Curing Process, the Relationship of Exposure Conditions and Measurement in UV Process Design and Process Control.
- [2] Augustine O. N. 2004. Development of the RIDFT Process Incorporation of UV Curing Technique. The Florida State University College of Engineering.



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- [3] Bolton J. R. 2005. Fundamental of UV Treatment. Terms and Definitions Do Matter.
- [4] Goin J. 2004. Anatoly Skirda, Eugene Tokhtuev, Understanding UV Monitoring for Air and Water UV Treatments.
- [5] Gutowski T. G. 1997. Advanced Composites Manufacturing. 2nd Edition. John Wiley.