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Effects of Process Parameters on Selected Properties of Liquid Compression-Molded Vinyl Ester Sheets

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ABSTRACT

Vinyl esters combine the best of polyesters and epoxies in terms of properties and processing. Without complicating presence of reinforcing fibres, this study investigated the effects of catalyst amount, preheating time, molding temperature, and pressure on flexural and water absorption properties of cast vinyl ester (VE) using a factorial experiment. Longer preheating time enhanced the stiffness of VE, while higher molding pressure reduced the flexural modulus. All the four factors did not affect the flexural strength and elongation at the break of molded VE significantly. Using a high molding pressure also caused molded VE to have higher water absorption for a long water exposure period. Meanwhile, greater water absorption at bigger amount of catalyst and higher preheating temperature indicate possible interactions between these factors. The results suggest possible negative effects of high molding pressure through the increase in the network of micro-cracks, and thus lowering the integrity of cast VE sheets. Judicious selection of the process parameters was required in order to obtain good quality molded VE sheets and by extension fibre-reinforced VE composites. Molded VE-unsaturated polyester (UP) blend is a significantly different material which is 1.49 times stronger, 2.38 times more flexible, but it is 0.69 less stiff than neat VE and with significantly higher water absorption. The results obtained warrant for a further investigation in process optimization of VE molding and the use of VE-UP blend as a matrix for natural fibre-reinforced composites.

Keywords: Vinyl ester, compression molding, factorial experiment, flexural properties, water absorption

INTRODUCTION

Thermoplastics and thermosets have been used as the continuous phase to bind the discontinuous fibrous or particulate phase in polymer composites. Thermosets are relatively easy to process and are well-suited for structural applications (Mallick, 1993). Though not directly recyclable, thermosets may still make positive environmental contributions through lower energy usage and air emissions (Joshi *et al*., 2004). Combining the best properties of epoxies and polyesters, VE resins are much less researched despite being tougher, more resilient, and less susceptible to water degradation by hydrolysis (Li, 1998). Nevertheless, there has been little use of VE with natural fibres like pineapple leaf fibres (PALF) (Arib *et al*., 2004) and more so of VE-UP blends.

The properties of the final composites depend on the individual properties of the matrix, fibre, and the nature of the interface between the two phases. Research has normally been carried out to understand either ingredient-structure-property relationships of resins or those of the fibre-resin combinations (Anon, 2008; Li, 1998). Therefore, there is a need to study the effects of processing

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parameters of liquid compression molding (LCM) on the cast matrix properties with the aim to optimize the process, particularly when fibre samples are scarce. LCM is seldom used to fabricate natural fibre-reinforced composites (Arib *et al*., 2004) and results have been varying, apart from the fact that it is limited to composite mechanical properties only (de Deus *et al*., 2005; de Sousa *et al*., 2004; Takagi and Asano, 2008; Mwaikambo and Ansell, 2003). As reported, molding pressure may not have any effect on the composite mechanical properties (de Deus *et al*., 2005) or even decrease them (Mwaikambo and Ansell, 2003).

Literature search indicates that there has been no previous work conducted on any simultaneously studying the effects of processing parameters on the mechanical and physical properties of molded VE and VE-UP blend. In this study, sheets of VE were compression-molded while varying the catalyst amounts, resin preheating time, molding temperature, and pressure. Their effects on the flexural and water absorption of this resin cast using LCM were also studied. Some comparative work was also done on VE-UP blend.

MATERIALS AND METHODS

Sample Preparation

The resins used were Bisphenol-A epoxy VE, Hetron 922, supplied by Act (UK) Ltd., whereas Synolac standard UP resin was supplied by Cray Valley Resins (M) Sdn. Bhd. Antonox-90 methyl ethyl ketone peroxide (MEKP) was used as the catalyst. VE was catalyzed with MEKP (*see* Table 1) and the mixture was stirred well before pouring it into the cavity of a flat three-piece mold and molded using a Technopress 40HC-B (Technovation) compression molding machine. Catalyzed resin preheating time, molding temperature, and pressure were varied as stipulated. Samples were molded for 10 minutes before cooling at 20°C without pressure and left to cure at ambient temperature for a minimum of 72 hours. Specimens were cut into required dimensions using a Pro-light Machining Centre (Light Machines Corp.). Sheet samples of VE-UP (50:50) blend were also press-molded.

TABLE 1
Processing parameters and their levels

Factor level	Catalyst (phr)*	Preheat time (minutes)	Molding temp. (°C)	Molding pressure (MPa)
Low	1.0	3	40	2.8
High	2.0	7	60	5.6
Centre-point	1.5	5	50	4.2

*phr – parts per hundred resin

Testing

Sample flexural properties were evaluated using an Instron 3365 tensile testing machine with a 5 kN load cell utilizing specimens of 63 mm long, 10 mm wide and 3 mm thick and ASTM D790 as a reference. In each case, five specimens with a span/thickness ratio of 16 and 2 mm/min test speed were used. Square specimens with 10 mm by 10 mm and 3 mm thickness were used to study water absorption as per ASTM D570. Samples were dried for 24 hours at 50°C in an air oven until constant weights were obtained. Conditioned samples were immersed in distilled water at ambient temperature. The samples were periodically taken out for weighing to the nearest 0.1 mg using

a Sartorius CP224S balance before they were immediately re-immersed. The specimens were carbon-coated using a Polaron SC7640 sputter coater before they were examined under a JEOL JSM – 5600 scanning electron microscope operated at 5 - 15 kV.

RESULTS AND DISCUSSION

The flexural properties and water absorption of the tested samples presented in Table 2 were analyzed following the examples in Wadsworth *et al*. (2002) and using Statsoft Statistica Release 7. The property change observed in optimizing matrix properties might turn out to be larger than those obtained by optimizing composite properties (Anon, 2008), and this would be valuable especially for composites with low fibre volume fractions. With only eight runs, effects of the four independent factors may be analyzed independently even though the isolation of interaction effects is not possible. The centre points chosen were not significantly different from the overall mean of all the points, and this suggested that the selected factors were linearly related to the dependent variables.

The calculations of student's t test and analysis of variance (ANOVA) of the results led the researchers to the following observations:

a. Increasing the catalyzed resin preheating time was found to significantly increase vinyl ester flexural modulus, while higher molding pressure significantly reduced the bending stiffness. On the other hand, the amount of MEKP and molding temperature did not affect flexural modulus. Though requiring further tests, it may be suspected that too high a molding pressure may affect the structural integrity of cast matrix and thus influence the mechanical properties especially the flexural modulus.
b. All four independent process parameters did not have any significant impact on the bending strength and elongation at the break of molded vinyl ester.
c. Increasing the molding pressure significantly increased the 432-hour water absorption of the molded vinyl ester. Though not significant, the trend might be observed in the case of 24-hour water absorption as well. Apparently, some finite amount of time was required for water molecules to diffuse through the networks of micro-crack and voids.
d. The average water absorbed by VE was 0.179% higher for 5.6 MPa molding pressure than for 2.8 MPa, representing a 32.8% increase. It was evident that water absorption was different between the two molding pressures. Though pressure consolidated the molding by significantly reducing the micro-crack network and voids in non-pressed moldings (*Fig. 2a*), too high a pressure might possibly reverse that effect and adversely affect the VE sheet by extension fibre-reinforced VE composites (Takagi and Asano, 2008; Mwaikambo and Ansell, 2003). Possible increase in the network of micro-crack could be indicated by the increase in water absorption.
e. Combining the high levels of MEKP and molding temperature (2.0% and 60^{o}C) led to the average amount of water absorbed 0.149% higher than the average difference between the catalysts over both molding temperatures (40^{o}C and 60^{o}C); a value not less significant than 0.179% given in (b) above (see *Fig. 2b*). The intersection in *Fig. 2b* indicated a possible strong interaction between catalyst amount and molding temperature, as highlighted by the ANOVA table in *Fig. 1*.

Molding of hybrid VE-UP (50:50) produced a significantly different material. The cast hybrid matrix was 1.48 times stronger and 2.38 times more flexible, but 0.69 less stiff than neat VE. However, its water absorption was substantially worse than that of the neat VE. This combination of properties may be suitable for applications requiring high toughness and may be further explored.

TABLE 2
Half-fractional factorial design with 2-level (2**(4-1)) standard design with the responses

Run	Factor				Response				
	Catalyst (phr)	Preheating time (min)	Molding temp (°C)	Molding pressure (MPa)	Flexural modulus (MPa)	Flexural strength (MPa)	EAB* (%)	24h water absorption (%)	432h water absorption (%)
1	-	-	-	-	2498.2	68.5	4.0	0.159	0.518
2	+	-	-	+	2352.9	54.1	3.2	0.226	0.652
3	-	+	-	+	2466.3	56.9	3.2	0.325	0.756
4	+	+	-	-	2778.6	52.2	2.7	0.192	0.495
5	-	-	+	+	2381.0	64.9	3.7	0.246	0.576
6	+	-	+	-	2598.2	57.5	3.2	0.270	0.651
7	-	+	+	-	2710.5	57.3	2.9	0.177	0.520
8	+	+	+	+	2371.9	59.4	3.2	0.273	0.917
9	0	0	0	0	2713.3	62.5	3.2	0.297	0.651
10	0	0	0	0	2674.3	57.3	3.0	0.183	0.571
11	0	0	+	+	1760.3	88.1	7.7	0.605	1.391

*EAB – extension at break

Effect Estimates; Var.:Var5; R-sqr=.97185; Adj:.87331 (Spreadsheet26)
2**(4-1) design; MS Residual=.0020926
DV: Var5

Factor	Effect	Std.Err.	t(2)	p	-95.% Cnf.Limt	+95.% Cnf.Limt	Coeff.	Std.Err. Coeff.	-95.% Cnf.Limt	+95.% Cnf.Limt
Mean/Interc.	0.630762	0.014466	43.60309	0.000526	0.568520	0.693004	0.630762	0.014466	0.568520	0.693004
(1)Var1	0.086516	0.032347	2.67462	0.115971	-0.052662	0.225693	0.043258	0.016173	-0.026331	0.112847
(2)Var2	0.072649	0.032347	2.24592	0.153787	-0.066529	0.211826	0.036324	0.016173	-0.033264	0.105913
(3)Var3	0.060601	0.032347	1.87348	0.201866	-0.078576	0.199779	0.030301	0.016173	-0.039288	0.099889
(4)Var4	0.179156	0.032347	5.53857	0.031087	0.039978	0.318333	0.089578	0.016173	0.019989	0.159167
1 by 2	-0.018095	0.032347	-0.55939	0.632181	-0.157272	0.121083	-0.009047	0.016173	-0.078636	0.060542
1 by 3	0.149440	0.032347	4.61992	0.043797	0.010263	0.288618	0.074720	0.016173	0.005131	0.144309
1 by 4	0.032350	0.032347	1.00009	0.422614	-0.106828	0.171528	0.016175	0.016173	-0.053414	0.085764

Var 1 - Catalyst Amount
Var 2 - Preheat Time
Var 3 - Molding Temperature
Var 4 - Molding Pressure

Fig. 1: Statistical R7 output of the main effects and interactions of the process parameters on 432h water absorption

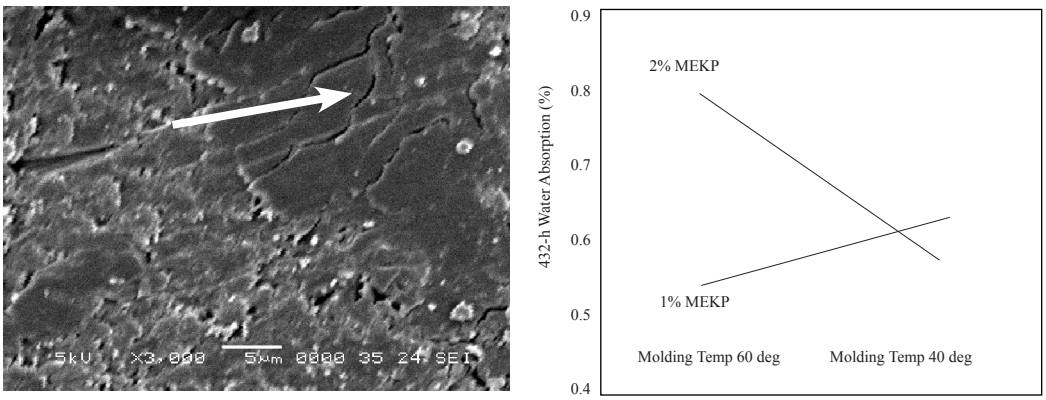

Fig. 2: (a) Microcrack network in the VE cast without pressure, and (b) interaction plot between catalyst amount and molding temperature

CONCLUSIONS

Based on the present study, it may be concluded that matrix optimization should be carried out not only to arrive at approximately optimal processing parameters before loading of reinforcement, the results may also provide clues to the resultant composite properties. Factorial experiments should be used as interactions of factors that may cause unsolicited significant property changes. Thus, further studies may be warranted on matrix optimization and the use of VE-UP blend as matrix for natural fibre-reinforced eco-composites.

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