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OPTIMIZATION OF TENSILE STRENGTH OF PULTRUDED GLASS FIBER COMPOSITE

Anurag Gupta[1], Dr. Hari singh[2], Dr. R.S. Walia[3]

[1]*Mechanical engineering Department, KIET, Ghaziabad & N.I.T Kurukshetra , (India)*

[2]*Mechanical engineering Department, N.I.T Kurukshetra Kurukshetra, Haryana, (India)*

[3]*Mechanical Engineering Department, D.T.U, New Delhi, (India)*

ABSTRACT

Pultrusion is the manufacturing process for producing fiber reinforced composites with constant cross sectional profiles comparatively at low cost. There are several parameters which affect the properties of pultruded glass fiber reinforced polymer (GFRP) composite. In the present work three parameters i.e. pulling speed, Die temperature and weight % of calcium carbonate in polyester resin as filler to optimize the tensile strength of glass fiber reinforced polymer (GFRP). Pultrusion of GFRP composite strip is done on indigenous pultrusion setup and in this paper Taguchi L9 orthogonal array is used for Design of experiment and ANOVAs is carried out for analysis of result. The indigenous pultrusion setup is used for manufacturing of pultruded and it was found by ANNOVA analysis that the all three selected parameter significantly affect tensile strength of GFRP composite strip. It has been also observed that as the sie temperature increases from 125^{0}C tensile strength decreases while tensile strength of pultruded GFRP first increases and then decreases with increase in Pulling speed and % weight content of CaCO3. The optimum level of all three parameter gives 383MPa tensile strength of pultruded GFRP composite.

Keywords: Pultrusion, Polyester Resin, $CaCO_3$, Pulling Speed, Die Temperature

I. INTRODUCTION

Pultrusion is a process through which high-modulus, lightweight composite structural members such as beams, truss components, stiffeners, etc., of constant cross-section are manufactured at low cost . A schematic view of the process set-up is shown in Fig.1. The reinforcement may be fiber glass, carbon fiber, aramid or any natural fiber. The reinforcement material is pulled and guided through a resin impregnation system which can be either open resin bath or resin injection chamber. The resin matrix used for impregnation of fiber may be thermoset resin like epoxy, polyester or thermoplastic resin. The fiber reinforcement is fully wetted out such that all the fibers are saturated by the resin. The reinforcements and the resin pass through a heated die once the resin impregnates the reinforcement material. Inside the heated die, the state of the resin gradually changes from liquid to solid because of the exothermic reaction of the thermosetting resin. The cured and solidified product is pulled via a pulling mechanism and cut in to required length.

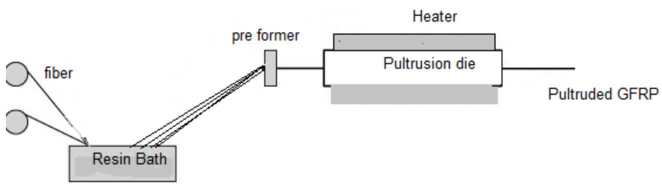

Fig.1 Schematic view of Pultrusion Process

Many researchers have investigated the effect of different pultrusion process parameter like pull speed, hot die temperature, fiber volume fraction and resin viscosity on mechanical properties of GFRP pultruded product. It was investigated by Cowen et al. [1] that with the increase of pulling speed the flexural strength and tensile modulus of elasticity both decreases. Vaughan et al. [2] have discussed in their work that the preheater temperature, pulling speed and cooling rate significantly affect the mechanical properties of pultruded composites. Ma et al. [3] varied fiber volume content and pulling speed to see the effect of these two process variable on mechanical properties for different glass material combination. Astrom et al. [4] concluded in his study only the pulling speed had significant influence on the flexural properties while preheated, heating die as well as cooling die temperatures do not affect significantly flexural properties of pultruded profiles. Chachad et al.[5] [6] proposed a three dimensional model of heat transfer and cure in pultrusion to characterize pultruded product by the desired mechanical properties or to realize a post-die shaping. A bi-dimensional finite element model was also proposed by Suratno et.al.[7] to simulate the influence of process parameters on GFRP pultruded composite rods. Wilcox et al. (1998) [8] analyzed the optimization of pultrusion process and found that the quality and

process pull force, position of peak exotherm as well as the impregnation duration.

efficiency of final product not only depend on parameters those set by the operator but also depends on the inter-process parameters that occur as a result of materials and process settings such as

Moschiar SM et al. [9] discusses that low temperature in heating die causes under curing and too high temperature causes smoldering of resin matrix . Joshi et al [10] [11] [12] discussed the resin shrinkage in GFRP pultruded composite and also proposed finite element/nodal control technique to optimize curing. Sarrionandia M et al. [13] concluded in his study that pulling speed depends on various conditions including the size of the pultruded profile, length heating die, die temperature and resin formulation. Two different computational methods, finite differences and elements, developed and critically analyzed to model the curing of pultruded profile under heated die by Carlone et.al [14] . Gupta et.al [15] have used fillers content of bagasse fiber, carbon black and calcium carbonate as pultrusion parameter and kept the pulling speed die temperature and fiber content to evaluate the tensile strength of pultruded glass fiber polyester resin composite. It has been observed from the literature review a most of the researcher have taken pulling speed, Die temperature and fiber content as process parameter to optimize the pultrusion process for high strength product and no paper have been found in which the resin composition is taken as process parameter. In the present paper filler content i.e Calcium carbonate % is used as process parameter along with die temperature and pulling speed by keeping the fiber content constant.

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II. EXPERIMENTAL SETUP AND METHOD:

The all the experiment have been performed on indigenously designed and developed pultrusion setup complete setup is shown in fig.2

The complete pultrusion setup is assemblred on H iron section as shown in fig. The main parts of pultrusion setup are

1. Performer: it is nothing but a cold die which give initial shape to the impregnated glass fiber so that resin impregnated fiber easily enters in to hot die and also squeeze the extra resin.

2. Hot Die: the hot die used in this set up is made of stainless steel and can be split in to two parts so that cleaning of die is easy. Hard chrome plating of 25 microns thick is done to avoid abrasion of die due to the pultrusion of hard filler particles and cured pultruded product. For heating the electric of 1400 Watt are used with temperature controller the thermocouple is fixed at parting line of the die.

3. Puller: The puller of the pultrusion setup consist the following

(i) 3 Φ A.C. motor of 1H.P power

(ii) Arrangement for controlling the speed of pultrusion

(a) A .C frequency drive for 1H.P motor

(b) 1:60 speed ratio Gear Box

(iii) Pulling Rollers

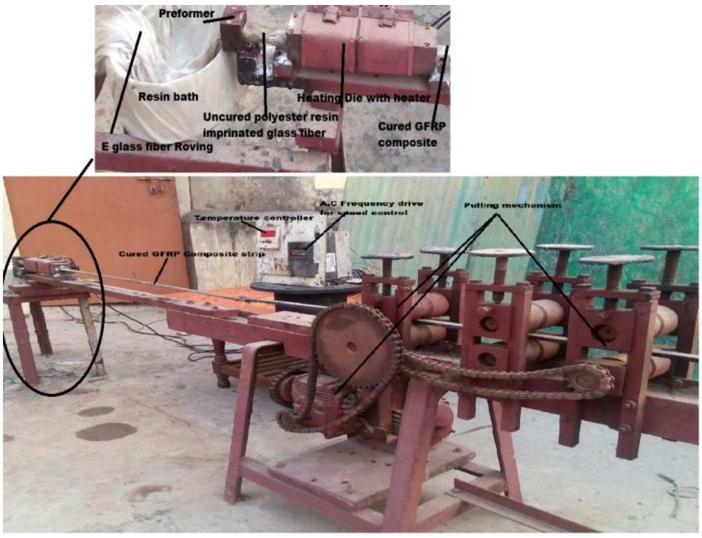

Fig.2 Indigenous Pultrusion Setup with Enlarged Heating Section

III. MATERIAL USED

1. Matrix: Unsaturated polyester resin is used as a.
2. Reinforcement: E glass fiber roving
3. Fillers: $CaCO_3$ of 100 µm particle size
4. Cobalt naphthelate of 6% concentration as accelerator

5. Methyl ethyl keton per oxide as Catalyst.

IV. EXPERIMENTATION

It was found in pilot experiments that the die temperature cannot be kept below 1100C because below this temperature even at minimum pulling speed possible in the setup i.e 50 mm /min complete curing do not take place. It was also found during pilot tests that the CaCO3 % more than 15% in resin compound produce problem in impregnation of glass fiber. The levels of three identified variable process parameter i.e die temperature, pulling speed, % of CaCO3 and constant parameter are given in Table 1.

Table1: Levels of Experimental Design Parameters

Levels		**1**	**2**	**3**
variable parameter	Die temperature(^{0}C)	125	150	175
	Pulling speed	**50**	**75**	**100**
	$CaCO_3$ (gm)	**5**	**10**	**15**
Constant parameters	% Catalyst	**1.2**		
	Fiber to resin ratio	**0.95to 0.96**		

Instead of using many glass fiber creel, glass fiber roving bundles of 1500 mm length and 180 to 185 g in weight are formed manually to achieve the thickness of composite so that experimentation cost can be reduced. The manually formed glass fiber roving bundles is shown in fig3. After the preparation of these bundles the die temperature and pulling speed of the pultrusion set-up was adjusted one by one for each experiment given in table 2. The filler CaCO3 in the particulate form was also properly mixed in the unsaturated polyester resin matrix according to the experiment run shown in table 2. The die temperatures and pulling speeds are set by temperature controller and A.C. Frequency drive respectively.

The roving bundles are wetted with resin filler compound by dipping into the resin bath and then pulled through a steel strip against the hot die; thus FRP strip of size 25x10x1500 mm is formed.

Fig.3: Fiber Bundle for Pultrusion

V. TESTING OF SPECIMEN

After manufacturing of all nine GFRP composite strips of 25x10x1500 mm according to the DOE, three specimen from each strip are formed according to ASTM D638 [16] as shown in figure4.

Fig. 4(a): Pultruded GFRP Composite Tensile test Specimen According to ASTM D638

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computerized universal testing machine manufactured by Fine Manufacturing Industry Miraj (Maharashtra), India is used for tensile tests according to ASTM D 638. The tensile test conducted and the specimens after tensile fracture are shown in

Figure. 4(b) and the results of tensile test are reported in table 2

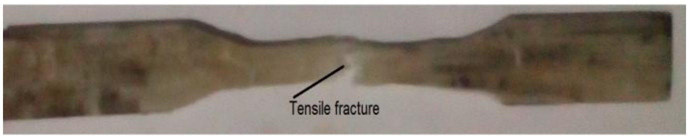

Fig. 4(b) Specimen after Tensile fracture

Table 2—Taguchi L_9 OA with responses (raw data and S/N ratios)

Run	Process parameters			Raw data (Tensile strength (MPa))			S/N Ratio (dB)
	Die Temp (^{0}C)	Pulling Speed (mm/min)	$CaCO_3$ (%)				
	Trial condition			R1	R2	R3	
1	125	50	5	290	295	295	49.35
2	125	75	10	388	380	381	51.66
3	125	100	15	257	255	252	48.12
4	150	50	15	334	333	340	50.52
5	150	75	5	306	303	308	49.70
6	150	100	10	245	247	246	47.82
7	175	50	10	268	266	268	48.54
8	175	75	15	302	305	301	49.62
9	175	100	5	285	287	289	49.16
Total				530.1	529.5	529.8	

R1, R2, R3 represent three repetitions of each trial; Grand average of tensile strength (T_{avg})= 297.25

The present study is carried out to analyse the effect of the various design parameters i.e. die temperature, pulling speed and % content of Caco3 with the help of L_9 Taguchi orthogonal array on tensile strength of pultruded GFRP profile.

The results from experiment runs were then converted into Signal-to-Noise (S/N) ratio. Usually, there are three categories of quality characteristics in the analysis of the S/N ratio,viz., the lower-the-better, the higher-the-better and the nominal-the-best. Taguchi recommends the use of S/N ratio to measure the quality characteristics deviating from the desired values. Regardless of the category of the quality characteristic, a greater S/N ratio corresponds to better-quality characteristic. Therefore, the optimal levels of the process parameters have the greatest S/N ratio. The tensile strength as response falls under the category of higher-the-better type and the S/N ratio for the same can be computed as [17] [18]:

$$(S/N)_{HB} = -10 \log\left[\frac{1}{R}\sum_{j=1}^{R}(1/Y_j^2)\right]$$

where Yj (j = 1, 2, ... n) are the response values under the trial conditions repeated R times. Analysis of variance (ANOVA) was performed to identify the process parameters that were statistically significant. With the S/N and ANOVA analyses, the optimal combination of the process parameters was predicted.

VI. RESULTS AND DISCUSSION

The average values of tensile strength and the S/N ratio for each parameter at level L1, L2 and L3 were calculated and are given in Table 3. These values have been plotted in Fig. 5 giving the trend of influence of process parameters on tensile strength at different levels of the process parameters.

Table 3 Average values and main effects of tensile strength of composite

	Level	Die Temperature			Pulling speed		$CaCO_3$
		Raw data	S/N ratio	Raw data	S/N ratio	Raw data	S/N ratio
Avg. values (tensile strength Mpa)	L1	310.33	49.71	298.78	49.47	280.67	48.93
	L2	295.78	49.35	330.44	50.33	335.22	50.45
	L3	285.67	49.11	262.56	48.36	275.89	48.79
Main effects (tensile strength MPa)	L2-L1	-14.56	-0.36	31.67	0.86	54.56	1.52
	L3-L2	-10.11	-0.24	-67.89	-1.96	-59.33	-1.66

L1, L2 and L3 represent levels 1, 2 and 3 respectively of parameters. L2–L1 is the average main effect when the corresponding parameter changes from level 1 to level 2. L3–L2 is the average main effect when the corresponding parameter changes from level 2 to level 3.

Table 4 Pooled ANOVA (S/N data)

Source	SS	DOF	V	P	F-Ratio	F-Ratio Table
Die Temp.	0.55	2	0.28	4.84	55.05	19
Pulling speed	5.81	2	2.91	50.78	577.87	19
% CaCO3	5.07	2	2.53	44.29	503.99	19
Error	0.01	2	0.01	0.09		
T	11.45	8		100		

*Significant at 95% confidence level

SS: sum of squares; DOF: degrees of freedom; V: variance; P: percent contribution

Table 5: Pooled ANOVA (raw data)

Source	SS	DoF	V	F-ratio	P	F-Ratio Table
Bagasse	2767.63	2	1383.82	6.39	149.09	3.49
Carbon Black	20771.19	2	10385.60	47.99	1118.96	3.49
$CaCO_3$	19558.74	2	9779.37	45.19	1053.64	3.49
Error	185.63	20	9.28	0.43	185.63	
T	43283.19	26	*	100		

*Significant at 95% confidence level

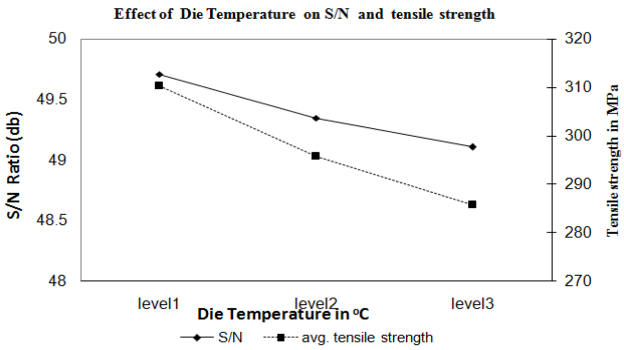

Figure5(a) response curve between Die Temperature level Vs S/N ratio and Tensile strength

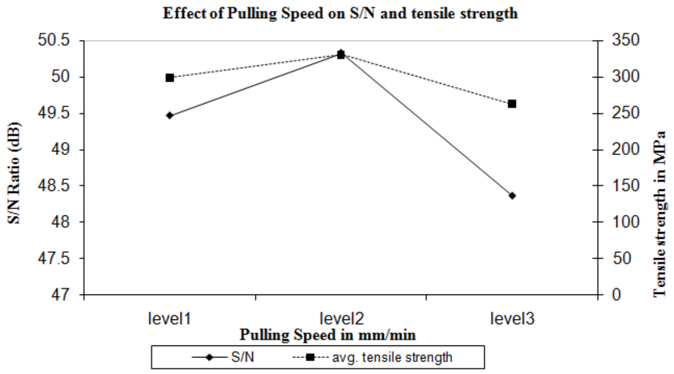

Figure5(b) response curve between Pulling Speed level Vs S/N ratio and Tensile strength

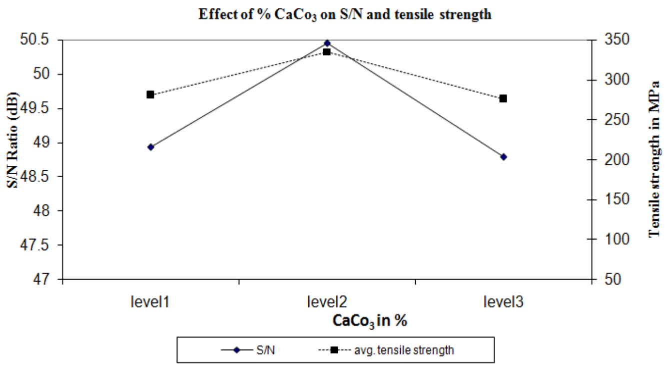

Figure5(c) response curve between %$CaCO_3$ level Vs S/N ratio and Tensile strength

Fig. 5—Effect of process parameters on tensile strength and S/N ratio (main effects) (a)Die Temperature (b) Pulling speed (c) % CaCO3 content size

ANNOVA analysis of the result shows that the three process parameter selected are significant and effect the tensile strength of the pultruded GFRP strip. Fig. 5(a) shows response curve between die temperature and S/N ratio and tensile strength. It is clear from the curve that as the temperature increases the tensile strength of the GFRP composite reduces. It was observed in the experiment run that proper curing of composite do not take place at low temperature i.e 125^{0}C and high pulling speed i.e 100mm/min on the other hand the matrix start burning and losing its binding strength at high temperature i.e175^{0}C and low pulling speed i.e. 50 mm/min.

Fig. 5(b) shows response curve between pulling speed and S/N ratio and tensile strength. As shown in the curve that the tensile strength first increases with the increase of pulling speed and then reduced with increase of pulling speed the reason behind this phenomena is that at low speed that the matrix exposed to heat for long time in the die and lose some of its binding strength as the unsaturated polyester resin is a thermoset resin while at very high speed the GFRP composite do not have sufficient time to cure in the heated die so the core of the composite do not cure properly.

Fig. 5(c) shows response curve between % Content of CaCO3 and S/N ratio and tensile strength. The curve indicate that the tensile strength of the composite increases as the % of Caco3 increases from 5% to 10% because initially the CaCO3 fills the voids in composite and help in better distribution of stress but further increase i.e. from 10 % to 15% the CaCo3 particles forms clusters and works as crack initiator and results in decrease in the tensile strength.

VII. ESTIMATION OF OPTIMUM TENSILE STRENGTH

The optimum value of tensile strength was predicted at the selected levels of significant parameters A1, B2 and C2 (Tables 3 and 5). The estimated mean of the response, i.e. tensile strength was determined as in Equation (1):

Tensile strength=TA1+TB2+TC2-2Tavg=381.50MPa .....(1)

Where Tavg: Overall mean of tensile strength = 297.25MPa (Table 2)

TA1=Average tensile strength at the second level of bagasse content (A2) = 310.33MPa

TB2=Average tensile strength at the first level of carbon black content (B1) = 330.44 MPa

TC2=Average tensile strength at the first level of CaCO3 (C1) = 335.22 MPa

The 95% confidence interval of confirmation experiments (CICE) and of population (CIPOP) was calculated by using the following equations:

$$CI_{CE} = \sqrt{F_a\ (1,\ f_e)V_e\left[\frac{1}{n_{eff}} + \frac{1}{R}\right]} \quad \text{.... (2)}$$

And

$$CI_{pop} = \sqrt{\frac{F_a\ (1,\ f_e)V_e}{n_{eff}}} \quad \text{... (3)}$$

where, Fα(1, fe) is the F ratio at the confidence level of (1 – α) against DOF 1 and error DOF fe, N is total number of results = 27 (treatment = 9, repetition = 3), R is sample size for confirmation experiments = 3,

Ve is error variance = 9.28 (Table 5),

fe = error DOF = 20 (Table 5).

$$\text{neff} = \frac{N}{1 + [DOF\ associated\ in\ the\ estimate\ of\ mean\ response]} = 3.86 \quad \text{... (4)}$$

F 0.05(1, 20) = 3.4928 (Tabulated F value) ... (5)

So, CICE = ±4.37 and CIPOP = ±2.9

The predicted optimal range (for a confirmation run of three experiments) is:

Mean tensile strength- CICE< tensile strength< Mean tensile strength + CICE : 377.13< tensile strength<385.62

The predicted optimal range for the population is as follows: Mean tensile strength – CI pop< tensile strength< Mean tensile strength + CI pop : 378.35< tensile strength<384.

The optimal values of process parameters for the predicted ranges of tensile strength are as follows:

First level of Die temperature (A1) = 125^{0}C gm

Second level of carbon black content (B2) = 75 mm/min

Second level of CaCO3(C2) = 10% of resin weight

Confirmation experiments

As the optimum setting s of the parameters are First level of Die temperature (A1) = 1250C gm, Second level of carbon black content (B2) = 75 mm/min and Second level of CaCO3(C2) = 10% of resin weight actually this is the second experiment run of the L9 Orthogonal array and the average value of tensile strength of GFRP composite is 383MPa which is within the confidence interval of the predicated optima of tensile strength.

VIII. CONCLUSION

The effect of three process parameters Die Temperature , Pulling Speed and % CaCO3 on tensile strength of the GFRP composite was investigated. The following conclusions can be drawn from the study:

(i) The tensile strength at the optimum levels of Die Temperature , Pulling Speed and % CaCO3 is 383.0MPa.

(ii) Experiments on pultrusion of GFRP composite test pieces confirm that as the die temperature increases from 1250C to 175 0C tensile strength decreases .

(iii) Experiments also exhibit that within the testing levels the tensile strength first increases for pulling speed 50mm/min to 75 mm/min but tensile strength decreases further increase in pulling speed upto 100mm/min.

(iv) It was also concluded that the tensile strength of GFRP composite increases with increase of CaCo3 % from 5 to 10% but decreases with further increase in % of CaCO3.

(v) The predicted optimal range for tensile strength is CI_{pop}: 378.35< tensile strength<384.

(vi) The 95% confidence interval of predicted mean for tensile strength is 377.13< tensile strength<385.62.

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