Matrix Digestion Test for Glass Fibre Reinforced Plastics Composites

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***Abstract*- Fiber Reinforced Plastic (FRP) composite materials have several unique features. Important among these are the properties of superior specific stiffness and specific strength, and high corrosion and fatigue resistance. In addition, they offer excellent tailorability. Due to these attractive properties the application of composites has spread to almost every engineering field like marine, automobile, aerospace etc. The aim of the paper is to identify a better method of preparing Glass Fiber Reinforced Composite Laminates. Most predominant method of preparing GFRP composite laminates is Hand Lay-up but t has its own redundant properties like more air voids, resin rich or resin starved areas. These flaws are tolerable when it comes to general applications but on the whole, strength properties get affected. Hence an attempt to identify a better method of preparing GFRP composite laminate by adopting rolling concept is tried upon. A test rig to roll laminates was designed and fabricated for this purpose and later on this was used to draw laminates of different reinforcements. The same type of reinforcements was used in the hand lay-ups also with a view to inspect the resin content in the two manufacturing processes. To assess the resin content there are two methods as per ASTM standards namely Acid Digestion test and Matrix Digestion test also called Bunsen burner test. The latter was opted because of its simplicity, ease of testing and Acid Digestion test is particularly for Kevlar Fiber reinforced laminates as this fiber gets burnt off in a Bunsen burner.**

***Index Terms*- Advanced Forming Process, Chop Strand Mat ,Matrix Digestion Test, Woven Fabric, Woven Roving**

I. INTRODUCTION

Accurate determination of resin to fiber ratio in a given composite is an important parameter as it directly influences the mechanical properties. Two commonly used methods for determining the resin content in a composite laminate are: Bunsen burner test and acid dissolving technique. The latter was opted because of its simplicity, ease of testing and Acid Digestion test is particularly for Kevlar Fiber reinforced laminates as this fiber gets burnt off in a Bunsen burner. A Bunsen burner with a gas connection was used for firing it and the test coupon was held in a string for completing the burning .The test specimens were prepared by using both hand lay-up and advanced forming process which is based on machine rolling technique. Different combination of laminate samples with requisite number of layers of matrix & reinforcements were fabricated for the testing purpose. Initial and final weight of the test sample with and without string was noted after burning . The difference of weight gives the fiber content which clearly gives the amount of resin in both the manufacturing techniques use.This experimentation clearly shows the accuracy of the thumb rule in preparing a resin mix in composite laminate preparation (two times resin weight to that of reinforcement), i.e., ideal volume of resin for a sound composite laminate with no air voids and proper wetting of the reinforcement as it is vital for the composite laminate strength to be realized.

This attempt is made to determine the ideal content of resin for a given composition of reinforcement and this has been reflected aptly in the graphs plotted for fiber content by weight values. Finally a comparison is made between the laminates manufactured using both hand lay-up and advanced forming in order to determine the more effective manufacturing technique. Advanced forming process is based on machine rolling which is increasingly becoming popular due to ease of manufacturing & even distribution of the matrix throughout the laminate. In this process, the rollers are either Teflon coated or Teflon sheet is used with the laminate. The laminate with the requisite number of layers of matrix & reinforcements is placed in a Teflon bag, which is open at one end. This Teflon bag with the laminate is passed through the rollers, which are of a required diameter. Due to

squeezing action of the rollers, the excess resin comes out of the laminate & the chances of air pockets in the laminate are very less. This laminate is placed over a cleaned glass plate applied with the release agent. This laminate is allowed to cure for 24 hours.

II. AIM OF THE STUDY

The aim of the present study is to evaluate the ideal content of resin for a given composition of reinforcement. To do the same, a comparative study between the commonly used hand –lay up method and an advanced forming method (machine rolling technique) is carried out to test that laminates prepared from which of these two methods will have the ideal content of resin for a given composition of reinforcement. Reinforcements of two different types of fibrers were prepared using both hand –lay up method and an advanced forming method (machine rolling technique).

To assess the resin content there are two methods as per ASTM standards namely:

1. Acid Digestion test
2. Matrix Digestion test also called Bunsen burner test.

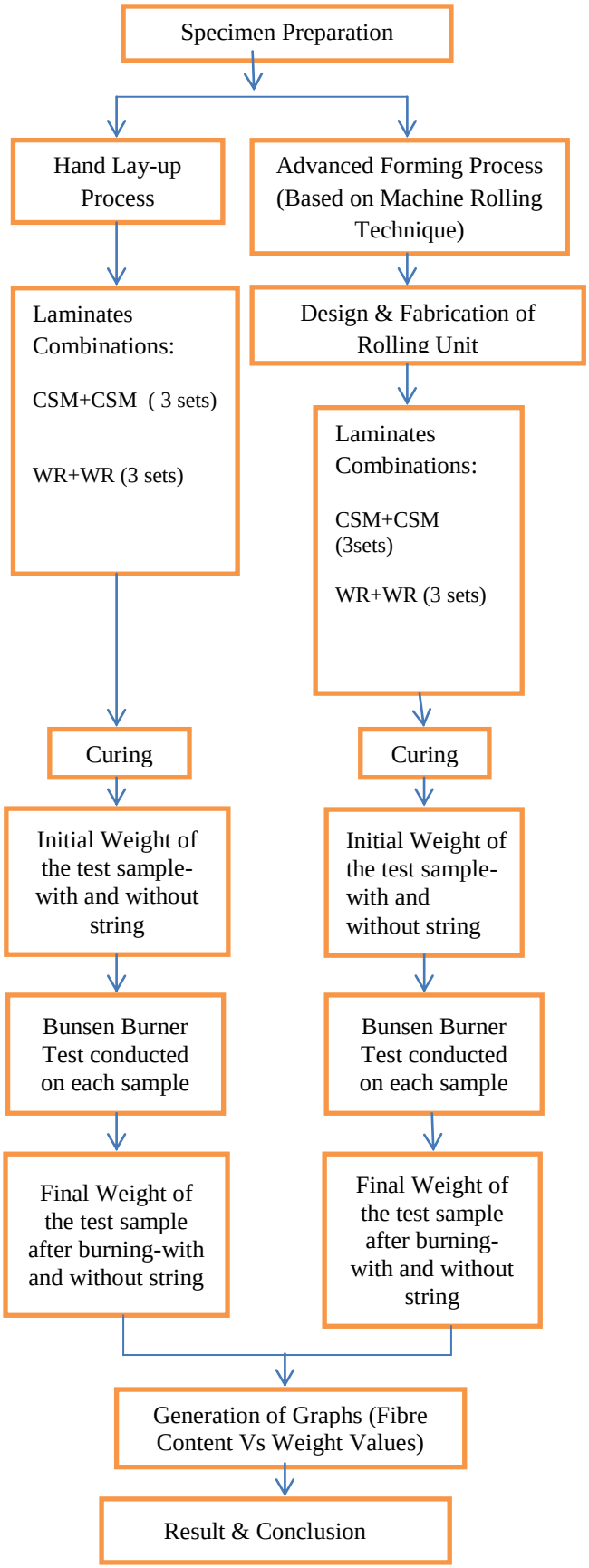
The latter was opted because of its simplicity, ease of testing and Acid Digestion test is particularly for Kevlar Fiber reinforced laminates as this fiber gets burnt off in a Bunsen burner. A Bunsen burner with a gas connection was used for firing it and the test coupon was held in a string for completing burning it. Initial weight of the test coupon with and without string was noted and after burning was noted. The difference of weight gives the fiber content which clearly gives the amount of resin in the two manufacturing processes. This experimentation clearly strikes home the point about the thumbs rule in preparing a resin mix in composite laminate preparation (two times resin weight to that of reinforcement), i.e., ideal volume of resin for a sound composite laminate with no air voids and proper wetting of the reinforcement as it is vital for the composite laminate strength to be realized.

The detailed methodology followed in carrying out this test is discussed in detail in the further sections.

III. METHODOLOGY

The sequence of operations in carrying out the matrix digestion test for the test coupons fabricated by both the hand –lay up method and an advanced forming method (machine rolling technique).
is explained in the flow chart below.

METHODOLOGY



IV. SPECIMEN PREPARATION USING HAND-LAY UP PROCESS

It may be noted that glass fiber mat and woven fabric are used in this study. General Purpose (GP) resin, twice in weight that of Glass fiber is measured. For this resin proportionate amount of accelerator i.e., Its 2% is added to form resin system. Later the lay-up table is prepared by cleaning its surface thoroughly. Its surface is waxed to give a fine smooth finish. Now the catalyst is added to the resin system forming resin mix, a layer of which is spread on the lay-up table using a resin brush. Chopped strand mat and Woven Fabric of 500mm x 200mm in size are laid one above the other along with resin mix. Care is taken not to misalign the layers one above the other. After the final layer is laid another glass pane cleaned to a smooth finish is laid on top and suitable weight applied and let set at room temperature for 24 hours.

The proportions of raw materials used in basic specimen preparation are:

a. Chopped Strand Mat, woven Fabric and
b. Resin = two times of the weight of mats.
c. Catalyst = 1 % of Resin
d. Accelerator = 1 % Of Resin

- Accelerator added to Resin and mixed thoroughly forms resin system.
- Catalyst should be added into the resin system just before using it.
- Basic cast size is 500mm x 200mm.
- From these casts 25mm x 25mm sized specimens are cut.
- For each cast, nine specimens were prepared.

The following tools are used for the specimen preparation

- Measuring jars
- Steel scale
- Resin brushes
- Glass rods
- Nylon roller
- Scissors
- Mylar sheets
- Glass plates
- Hack saw

In the hand lay-up process, a liquid material, normally polyester resin is combined with glass fibers (in the form of surface mat, chopped strand mat and/or woven roving and glass cloth). A chemical reaction is initiated in the resin by means of a catalyst and accelerator, which causes the resin to harden into a strong light final part in which the resin serves as the substrate and the fibers as the reinforcements. The glass fibers and resin are supplied sequentially on the mould to build the required thickness. The entrapped air being removed by rollers or by proper stroking with the brush. If a high quality surface is desired a gel coat is applied to the mould prior to lay-up normally cures at room temperature but heat may be $u_0$ed to accelerate cure. Initially releasing agent like wax is applied on the working table for suitable releasing of films. Almost any shape can be made by the hand lay-up process and is controlled by the ability of the fiber reinforced to conform to contours.

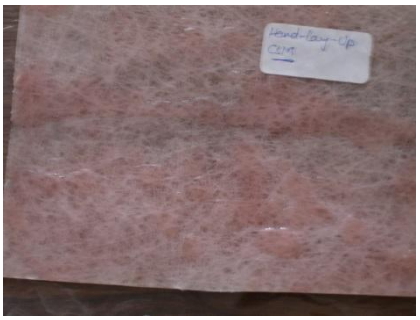


Fig.1 Hand lay-up sample specimen

V. SPECIMEN PREPARATION USING ADVANCED FORMING PROCESS

4.1 Fabrication of Rolling Unit for advanced forming process:

The Design and fabrication of rolling unit is based on the machine rolling technique of manufacturing laminates. The specifications and design parameters of the rolling unite is as given below.

Specification of Rolling Unit:

- Width of the roller 220 mm
- Diameter of the roller 111.4 mm
- Weight of roller 5kg each (two rollers)
- Speed of the roller 15rpm
- Number of roller used 2
- Upper roller (dead roller) adjustable
- Bottom roller moving type
- Type of bearing used ball bearing
- Material of the roller is made up of cast iron

4.2 Steps for fabricating the roller:

- Roller is made of cast iron, 2 cylindrical solids of more than 111.4 mm in diameter and more than 500 mm in length are used
- The metal is subjected to turning operation in lathe; turning is done to a diameter of 111.4 mm exactly at the middle of length 220 mm.
- The end sides are maintained at diameter 25 mm for a length of 140 mm including the clearance from ends of each side i.e. the shaft has to fit inside the bearings.
- The edges of roller are chamfered so that the load concentration will be distributed uniformly.
- The roller and shaft edges are made round to the rigidity of the joint.
- The roller circular surface is subjected to heat treatment to obtain the hard and very smooth surface .The shaft circular surface is pressed, so that the shaft fits tightly inside the bearing.
- Ball bearings (rolling contact) bearings made up off high carbon steel are used for the easy rotation of the rollers. Since the rolling frication is very less in this type compared to the sliding contact bearings, hence it is called as anti-frication bearings.
- Bearings are fixed at the roller ends, which also carry the roller load.

Fig.2 Front View of Rolling Unit

Fig.3 Side View of Rolling Unit

4.3 Fabrication of sample laminates.

Fig-4 Rolled Laminates

VI. MATRIX DIGESTION TEST

In this test a Nichrome wire is wound around the specimen and weighed. The specimen with the wire is kept over the Bunsen burner for about 2-3 minutes, so that the resin is burnt completely by leaving only the fibers. The fiber is then cooled and weighed. cool the fibers and weigh them. Fiber content by weight W_f is computed by using the following formula;

$W_f = [W_1-(W_2-W_3)]/W_1$

Where : W_1=weight of the composite specimen

W_2=weight of the specimen and wire

W_3=weight of the fiber and wire

Fiber content by volume is given by

$V_f=W_f [W_f+p_m(1-W_f)/p_f] –1$

Where p_m=mass density of matrix (resin)

p_f =mass density of carbon fiber

Fig-5 Test coupons from 3 sections of laminate

Fig-6 Burnt sample

VII. HAND LAY-UP TEST DATA

A set of three hand lay-up samples were made for each of the laminate combinations i.e

1. CSM+CSM (1,2&3sets)
2. WF+WF (1,2&3sets)

9 test coupons from the three sections of the laminate sheet were selected from front, middle and back portion respectively. The values obtained for the laminate combinations with respect to W_1, W_2,W_3 & W_f is calculated using the formula mentioned in the above section. The values of laminate combination CSM+CSM (all 3 sets) are shown from table 1-3 respectively.

Table 1-Hand Lay-up CSM+CSM (set 1) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	1.8856	2.6275	1.3720	0.3342
	2	1.9238	2.5948	1.2110	0.2807
	3	1.7613	2.4189	1.3025	0.3662
Middle	4	1.5262	2.1722	1.1548	0.3334
	5	1.4020	1.9870	1.1572	0.4081
	6	1.5971	2.2220	1.1161	0.3076
Back	7	1.5876	2.1728	1.2130	0.3954
	8	1.5459	2.1626	1.1694	0.3575
	9	1.5568	2.2228	1.1848	0.3332

Table 2-Hand Lay-up CSM+CSM (set 2) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	1.2481	1.9125	1.2068	0.4346
	2	1.2185	1.8967	1.2506	0.4698
	3	1.3356	1.8993	1.1301	0.4241
Middle	4	1.2521	1.8189	1.0461	0.3828
	5	1.1165	1.7780	1.1654	0.4513
	6	1.2586	1.8730	1.0951	0.3819
Back	7	1.1976	1.8047	1.1688	0.4690
	8	1.0894	1.7218	1.1773	0.5002
	9	1.5336	2.1556	1.2306	0.3968

Table 3-Hand Lay-up CSM+CSM (set 3) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	1.6085	2.2413	1.1203	0.3031
	2	1.9626	2.4125	1.4053	0.4868
	3	1.8529	2.6569	1.2354	0.2328
Middle	4	1.6815	2.2863	1.1784	0.3411
	5	1.6912	2.2761	1.1850	0.3548
	6	1.9908	2.6396	1.3785	0.3665
Back	7	1.5820	2.0930	1.0649	0.3501
	8	1.7060	2.2767	1.1797	0.3570
	9	1.5335	2.2095	1.2350	0.3645

The values of laminate combination WF+WF (all 3 sets) are shown from table 3-6 respectively.

Table 4-Hand Lay-up WF+WF (set 1) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	1.1198	1.6989	1.1645	0.5228
	2	1.1101	1.7678	1.2462	0.5301
	3	1.1318	1.8250	1.2966	0.5331
Middle	4	1.0782	1.7129	1.1979	0.5223
	5	1.0884	1.7143	1.1824	0.5113
	6	1.1054	1.8159	1.2891	0.5234
Back	7	1.0662	1.6632	1.1772	0.5442
	8	1.0437	1.7371	1.2523	0.5355
	9	1.0931	1.8343	1.3207	0.5301

Table 5-Hand Lay-up WF+WF (set 2) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	0.9391	1.7006	1.3004	0.5738
	2	0.9258	1.5363	1.1474	0.5799
	3	0.9312	1.7447	1.3416	0.5639
Middle	4	0.9471	1.6031	1.1987	0.5730
	5	0.9631	1.5946	1.1725	0.5617
	6	0.9337	1.5590	1.1449	0.5565
Back	7	0.9552	1.5984	1.2034	0.5865
	8	0.9030	1.5228	1.1421	0.5784
	9	0.9574	1.6922	1.3095	0.6003

Table 6-Hand Lay-up WF+WF (set 3) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	1.1007	1.7765	1.2655	0.5357
	2	1.0945	1.7723	1.2406	0.5142
	3	1.0223	1.7217	1.2332	0.5222
Middle	4	1.1138	1.7390	1.2166	0.5310
	5	0.9633	1.7566	1.3167	0.5433
	6	1.0059	1.6934	1.2047	0.5142
Back	7	0.9735	1.9082	1.4716	0.5515
	8	0.9990	1.6681	1.1924	0.5238
	9	1.0222	1.6813	1.1837	0.5132

VIII. ADVANCED FORMING TEST DATA

A set of three advanced formed samples were made for each of the laminate combinations as mentioned above. 9 test coupons from the three sections of the laminate sheet were selected from front, middle and back portion respectively. The values obtained for the laminate combinations with respect to $W_{1,}$ $W_{2,}W_3$ & W_f is calculated using the formula mentioned in the above section. The values of laminate combination CSM+CSM (all 3 sets) fabricated by advanced forming process are shown from table 7-9 respectively.

Table 7-Advanced Forming CSM+CSM (1) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	2.0969	2.8518	1.5232	0.3664
	2	2.3066	2.9091	1.4498	0.3673
	3	2.1394	2.7081	1.3732	0.3760
Middle	4	2.2926	3.0280	1.5798	0.3683
	5	2.4715	3.1275	1.5194	0.3440
	6	2.4772	3.3901	1.7467	0.3366
Back	7	2.4578	3.0539	1.6738	0.4385
	8	2.5205	3.2809	1.4685	0.2809
	9	1.7446	2.3737	1.2423	0.3515

Table 8-Advanced Forming CSM+CSM (2) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	2.1199	2.7045	1.3758	0.3732
	2	2.2070	2.8883	1.4683	0.3566
	3	2.2208	2.7914	1.3747	0.3621
Middle	4	2.2878	2.9232	1.4642	0.3623
	5	2.5256	3.2090	1.5941	0.3606
	6	2.3809	3.0301	1.5084	0.3609
Back	7	2.6286	3.3926	1.7533	0.3764
	8	2.6257	3.3175	1.6532	0.3661
	9	2.7080	3.3018	1.5360	0.6003

Table 9-Advanced Forming CSM+CSM (3) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	2.3665	3.0626	1.4961	0.3380
	2	2.4306	3.1162	1.5534	0.3570
	3	2.4297	3.2186	1.6735	0.3640
Middle	4	2.0651	2.7345	1.4392	0.3728
	5	2.4596	3.1138	1.5114	0.3485
	6	2.5563	3.2406	1.5824	0.3513
Back	7	2.2912	2.9487	1.4150	0.3306
	8	2.6441	3.3309	1.6002	0.3454
	9	2.4064	3.0703	1.5224	0.3567

The values of laminate combination WF+WF (all 3 sets) are shown from table 10-12 respectively.

Table 10- Advanced Forming WF+WF (Set 1) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	0.7231	1.4068	1.2447	0.7758
	2	0.7821	1.4623	1.2733	0.7583
	3	0.7227	1.4734	1.2537	0.6960
Middle	4	0.9000	1.5786	1.2512	0.6362
	5	0.9407	1.6497	1.2886	0.6161
	6	0.8971	1.4692	1.3127	0.8255
Back	7	0.8424	1.5019	1.3063	0.7678
	8	0.7622	1.4958	1.3182	0.7670
	9	0.7231	1.3925	1.2033	0.7383

Table 11- Advanced Forming WF+WF (Set 2) test data

Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	0.7411	1.3924	1.3029	0.8792
	2	0.7515	1.2606	1.1340	0.8315
	3	0.7762	1.3826	1.2031	0.7691
Middle	4	0.7416	1.4274	1.2338	0.7389
	5	0.7572	1.4262	1.2045	0.7072
	6	0.6926	1.3341	1.1729	0.7672
Back	7	0.7201	1.3722	1.2369	0.8121
	8	0.8021	1.4562	1.3343	0.8480
	9	1.1774	1.7436	1.5001	0.7932

Table 12- Advanced Forming WF+WF (Set 3) test data

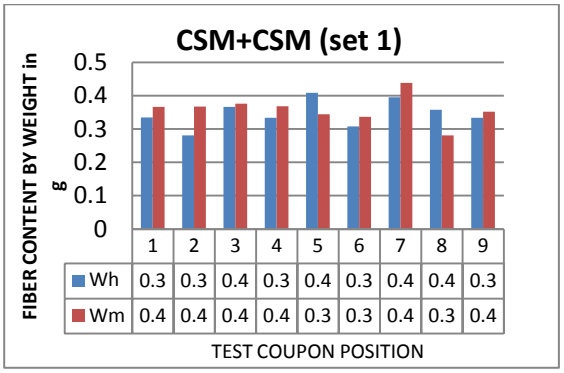
Position	#	W1 in g	W2 in g	W3 in g	Wf in g
Front	1	0.7324	1.2725	1.0651	0.7168
	2	0.7488	1.3121	1.1115	0.7321
	3	0.6397	1.2080	1.0739	0.7903
Middle	4	0.9286	1.5456	1.2415	0.6725
	5	0.7358	1.4108	1.1734	0.6773
	6	0.8332	1.3256	1.1445	0.7826
Back	7	0.8307	1.3877	1.1512	0.7153
	8	0.7850	1.4057	1.1884	0.7232
	9	0.7526	1.4270	1.2373	0.7479

IX. GRAPHS AND DISCUSSIONS

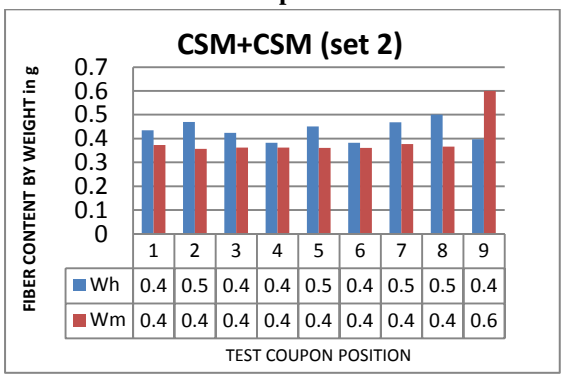
Graphs were plotted against the test coupon positions i.e from 1-9(as shown in fig-5)and the fiber content by weight values of both Hand lay-up and advanced forming laminates.

Graph 1-3 shows the variation in fibre content by wiegth factor in both Hand lay-up and advanced forming for CSM+CSM laminate.

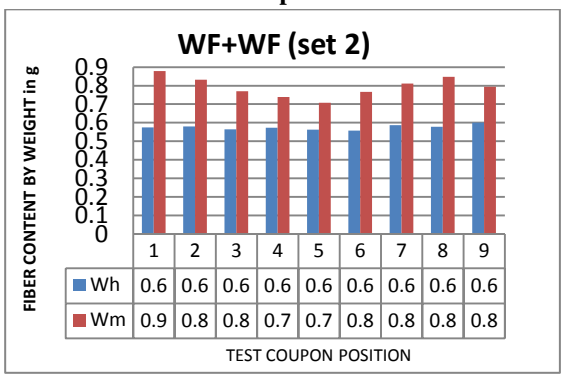
Graph-1



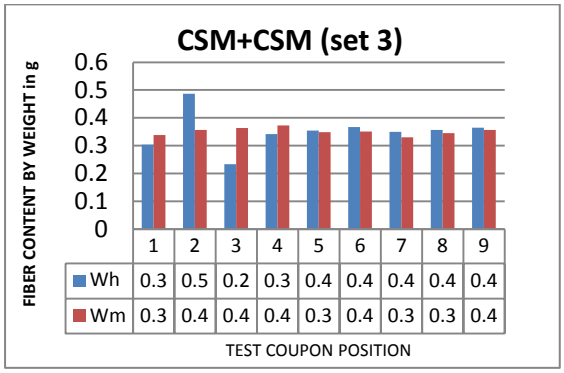
Graph-2



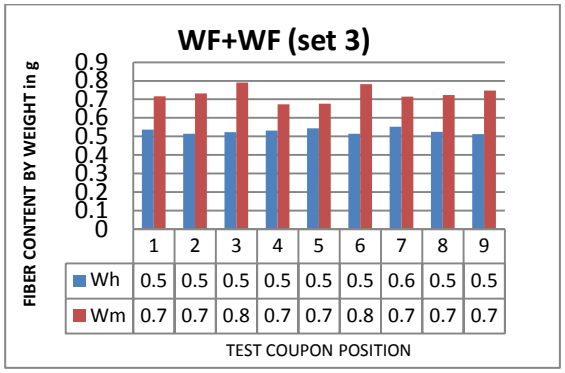
Graph-5



Graph-3



Graph-6

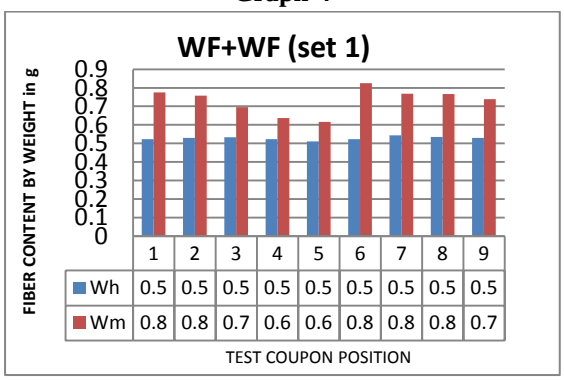


Discussion of Graphs 1-3: Chopped Strand Mat has strand loosely reinforced hence has compaction problems in specimen preparation.

From the matrix digestion test results it is evident that in hand lay-up coupons, the fibre content in different locations of given specimen varies which indicates uneven distribution of matrix, but in all advanced formed specimens the test coupons show almost same fiber content which means matrix distribution is ideal in advanced formed specimens.

Graph 4-6 shows the variation in fibre content by wiegth factor in both Hand lay-up and advanced forming for WF+WF laminate.

Graph-4



Discussion of Graphs 4-6: Woven fabric has a small bunch of glass strands properly woven than woven roving. These results in better compact assemble of reinforcement which facilitates better laminates.

From the matrix digestion test results it is evident that the fiber content by weight in Advanced forming specimens in different location on same specimen is on the higher side as compared to hand lay-up.

Hence it is evident that the ideal distribution of matrix is better in advanced forming process than hand lay-up process, also it is clear that though CSM has loosely reinforced strands specimen preparation becomes difficult, but still in advanced process it is more effective than hand lay-up. Whereas woven fabric on the other hand is more effective in hand lay-up than advanced forming process. To overcome these constraints hybrid laminates with combinations like CSM+WF, CSM+ WR, WR+WF can be tried for further tests.

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