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# Accelerated testing for long term durability of PU Foam Cored E-Glass reinforced vinyl ester sandwich composites

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### ABSTRACT

The prediction of long term mechanical properties of sandwich structures consisting of PU foam core and Vinyl Ester (VE) / Glass face sheet material under temperature, humidity and salt fog environment were performed by accelerated testing methodology based on the Time-Temperature superposition principle (TTSP). PU foam cored VE/ glass fibre reinforced sandwich structures with varied densities of PU foam were prepared and tested for degradation in flexural properties at three different temperatures (30° C, 40° C, and 50° C) and RH 95% for a period of 120 days. The degradation in flexural properties such as Facing Bending Strength (FBS), Core Shear Strength (CSS) and compression properties (edgewise and flatwise strength) were studied. The master curves for FBS, CSS, ECS and FCS were constructed by using the test data obtained from the degradation studies at three different temperature conditions based on Time temperature super position principle and the acceleration factor has been determined. An effort has been made to develop methodologies for accelerated testing of sandwich structures for long term durability considering time, marine ambience, temperature and humidity as major influencing parameters

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### Introduction

Durability of a material or structure is defined as its ability to resist cracking, oxidation, chemical degradation, delamination, wear, and/or the effects of foreign object damage for a specified period of time, under the appropriate load and environmental conditions [1]. Durability analysis of materials is vital before a new product is being incorporated into industry. Long term durability of sandwich composites under aggressive service environments like temperature, humidity, sea water, corrosive environment etc. is a primary requirement especially when these materials are to be used under marine ambience. However, it is impractical to do real time testing under these conditions as it requires long time for measurements.

Hence accelerated testing methodologies have been applied to study the long term durability of composites. Existing accelerated testing methodologies used for metals cannot be applied to composite materials, since the methodologies are not suitable for viscoelastic materials such as polymeric composite materials, which exhibit strong time and temperature dependence.

Accelerated testing methodology based on time temperature superposition principle (TTSP) has been proposed for predicting the long term durability of polymer composites. This principle was originally developed for non destructive material properties, but recent studies have shown that it can also be applied to determine the failure properties of the composite materials. [2-12]. Extending this principle to sandwich composites, in the present study a methodology has been developed to predict the compression and flexural properties of sandwich composite materials. Elevated temperature condition is used to accelerate the mechanical degradation.

TTSP is based on the assumption that the process involved in molecular relaxation or rearrangement in viscoelastic materials occur at accelerated rates at high temperatures and that there is a direct equivalence between time and temperature [13]. Therefore the time over which these processes occur can be reduced by conducting the measurement at elevated temperatures and shifting the resultant data to lower temperatures. The result of this shifting is a master curve where the material property of interest at a specific temperature can be predicted over a broad time scale.

In order to stimulate accelerated aging, the exposure can be intensified by providing a higher temperature, a higher saline concentration, or a higher amount of fog. In the alkaline corrosion research performed by Porter et.al [14], the acceleration is accomplished by exposing the glass fibres to high pH values and immersing the fibers in a bath at an elevated temperature. It has been shown that seawater immersion and salt-fog exposure are the main causes of degradation of glass fiber reinforced plastics used in naval applications [15]. In the present work salt-fog exposure test is conducted, as the previous studies have shown that it is one of the most aggressive exposures.

### Accelerated Testing

The only way to test the durability of composite materials in order to incorporate them quickly into infrastructure is accelerated testing, since 50-70 years of performance data is not available for these newly developed materials. Therefore, first understanding the failure mechanisms of FRP products and second, developing accelerated test procedures for predicting service life are vitally important for the selection of FRP products. The testing methodology that shortens the duration of the degradation process by some acceleration factors and uses the short-term measurements in the prediction of material's service lifetime is called accelerated testing.

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Accelerated tests have two phases. In the first phase, test materials are exposed to different environmental conditions that have one or more acceleration factors. These factors may be the use of high temperatures, mechanical loads or chemicals. During conditioning of the specimens, a set of measurements is made to record the changes in the mechanical, physical, thermal, or chemical properties of the materials over time. These changes are used as an indicator of the amount of physical and chemical aging that has taken place due to accelerated conditions. The second phase of accelerated testing is the long-term prediction of the mechanical or physical properties. In this stage, a predictive methodology that uses the accelerated test data is developed using degradation models. These degradation models should account for the combined effects of several factors and different changing service conditions through the service life of the materials for realistic predictions. Unfortunately, such models have not been developed yet. However, some scholars have proposed relatively successful degradation models through their experimental work. The test plan proposed for accelerated testing of glass fibre reinforced polymer matrix composites has been used in the present study for the accelerated testing of the sandwich composites [16-17].

#### Acceleration Factor

In all accelerated tests, an acceleration factor has to be chosen to accelerate the degradation of the tests specimens. Typical accelerating factors are; mechanical load (including vibration and shock), voltage, current, temperature (including thermal cycling and shock), weathering (ultraviolet, radiation and humidity) and the use of high concentrations of chemical environments. Dual acceleration factors can also be applied but this requires a more elaborate methodology. So it is not commonly preferred. It is very important that the decision on the acceleration factors be based on an analytical model, which is going to be used in the second step. Temperature is the most common acceleration factor for the materials used in naval applications.

#### Degradation models for Service Life time Prediction

The analytical models established to relate the degradation to the variables involved (temperature, salt composition etc) under the application of a constant acceleration factor are:

i) Constant rate model ii) Power Law Model, iii) Exponential Model iv) Eyring Model and v) Arrhenius Model. Among these degradation models, the model using the Arrhenius concept is the most popular for naval applications to study the degradation mechanism as a function of temperature. Therefore Arrhenius steady-state temperature acceleration model is discussed in detail in this section.

This model can be used where temperature is the only significant accelerating factor and chemical reactions are the dominant concern [18]. The Arrhenius degradation rate can be written as in Equation 1 [19].

$$\beta' = A' \exp\left(\frac{-E}{k.T}\right) \text{----- [1]}$$

Where:

$\beta'$ : the Arrhenius degradation rate

E: the activation energy of the chemical reaction in electron volts.

k: the Boltzmann's constant,  $8.6171 \times 10^{-5}$  electron volts per ° C

T: the absolute Kelvin temperature.

$A'$ : a constant of the test conditions and product failure.

The acceleration factor for this model can be written as in Equation 2 to relate degradation in the accelerated environments to one in the service condition [19].

$$k = \frac{\tau}{\tau'} = \exp\left[\left(\frac{E_n}{k}\right)\left(\frac{1}{T} - \frac{1}{T'}\right)\right] \text{----- [2]}$$

Where:

k: Acceleration factor

$E_n$ : Activation energy for damage mechanism and material.

$\tau$ : Lifetime at reference temperature T (° K)

$\tau'$ : Lifetime at elevated temperature T' (° K)

k: Boltzman constant

Equation 2 can be used to monitor the changes in the performance of the FRP materials under the effect of moisture diffusion. However, it is very important to note that the activation energy will be different for each damage mechanism and material [18].

#### Materials and Methods:

##### Materials

The sandwich specimens used in the present study comprise of four different grades of E-glass fabrics (supplied by Vetrotex /Saint Gobian, India) in vinyl ester resin supplied by Ecmas, Hyderabad and three varied densities of polyurethane foam (PUF) core supplied by polynate Foams Pvt. Ltd. Bangalore. The sandwich specimen face sheet is synthesized using 2% Cobalt Octate accelerator, Methyl Ether Ketone Peroxide (MEKP) as promoter and Di Methyl Acetamide (DMA) as catalyst in Vinyl ester system. The fiber to resin volume ratio is maintained as 65:35. The samples are cured at room temperature for 24 hours followed by 70°C in oven for post curing. The sandwich specimen's specifications and various configurations used in the experiment are presented in Table 1.

**Table 1: Sandwich Composites - Specifications**

Sandwich Type	Resin	Fabric Type (E-Glass)	Core Material	Core Density(Kg/m <sup>3</sup> )
WR		Woven Roving – 360 gsm		
CSM	Viny lester	Chopped Strand Mat-360 gsm	PU Foam	100 - 300
SBM	3 mm	Stitch Bond Mat -610 gsm	(24 mm thickness)	
CSM (S)		Chopped Strand Stitch Mat- 420 gsm		

**Methods**

**Salt Fog Exposure and testing procedure**

The salt fog chamber (Fig.1) of size 0.566 m<sup>3</sup> (M/s CM Environ systems, Bangalore) was used to expose the specimens to salt fog as per ASTM B 117. The salt solution atomizes into a fine mist in the chamber. Hot and humid air is created by bubbling compressed air at 103.4 KPa through a tube which is around 3/4 full of hot (usually 48° C) deionized water. The salt solution is moved from the 60-gallon (0.227 m<sup>3</sup>) holding tank to the nozzle by a gravity feed system using a float switch and a plastic solenoid. When the hot, humid air and the salt solution mix at the nozzle, the latter is atomized into a corrosive fog. The chamber is usually heated during this cycle at 35° C by the chamber heaters. Fog distribution is controlled by the Uni-fog dispersion system. The specifications of the chamber are: temperature: -20° C to 85° C, humidity: 0 - 95 % RH with provision for salt spray.



**Figure 1. Arrangement of sandwich specimens in salt fog chamber**

The cut and edge sealed specimens (specified for Flexural, Edge wise compression and Flat wise compression) were exposed to 30° C, 40° C and 50° C temperatures and humidity of 95% RH for a maximum duration of 120 days.

Each test at a specific temperature was performed over a period of four months. The test temperatures selected for study are 30° C, 40° C and 50° C .These temperatures are well below the glass transition temperature /degradation of foam and vinyl ester. Specimens were removed at one-month intervals. The properties like core shear strength, Facing bending strength, Edgewise and flat wise Compression strength were measured for each temperature. The log-log plot of property versus time is plotted for each temperature. Each curve is shifted to 30° C, chosen as reference temperature and master curve is obtained. Master curve is the superimposition of the individual curves shifted onto the reference curve. The obtained master curve will give the amount of degradation as a function of time.

**Flexural testing**

The flexural strength of the PU foam cored, E-glass reinforced vinyl ester sandwich composites was determined for different immersion times using the three-point bending test approach. Flexural test aims at determining facing bending strength (FBS) and core shear strength (CSS) using the specimens of 127 mm length, 65 mm width and 30 mm thickness under ambient conditions using a 10 ton universal testing machine (Kalpak, Pune) with a strain rate of 2 mm per minute. The facing bending strength of the sandwich composite was calculated using the equation (4).

$$\sigma = \frac{PL}{2t(d+c)b} \text{----- (4)}$$

where  $\sigma$  is the facing bending strength (MPa), P is the maximum load (N), L- Specimen length; t is the face sheet thickness (3 mm), d- sandwich thickness (30 mm), c- core thickness (24 mm) and b- breadth of the sandwich specimen. The core shear strength of sandwich composites was calculated using equation (5).

$$\tau = \frac{P}{(d+c)b} \text{----- (5)}$$

where  $\tau$ - is the core shear strength (MPa),

The average values of FBS and CSS of three sandwich composite samples tested for each immersion time was considered.

**Compression testing**

**Flatwise & Edgewise Compression Testing:**

The flatwise compression test of the sandwich composites was carried out in accordance with ASTM 364 and edgewise compression test according to ASTM-365 standard. The specimen size in case of flatwise testing was 30x30x30 mm and for edgewise testing was 75x60x30mm. The flatwise and edgewise compression strength are calculated using equations (6) and (7) respectively.

$$FCS = \frac{P}{A} \text{----- (6)}$$

$$ECS = \frac{P}{A} \text{----- (7)}$$

where, P is the ultimate load (N) and A is the area of the facings.

The average values of FCS and ECS of three sandwich composite samples tested for each immersion time was considered.

**Results and Discussion**

**Degradation in Flexural Strength (CSS &FBS)**

All the composite specimens showed degradation in flexural strength, due to hygrothermic conditioning. The decrease in CSS and FBS values of sandwich composites as a function of PU foam core density and operating temperature are shown in **Fig. 2** and **Fig. 3** respectively. The flexural strength decreases with the increase in temperature. The drop in CSS and FBS is found to be more in low density composites. The degradation in CSS and FBS is highest for SBM sandwich composites and lowest for CSM-S sandwich composites. The drop in CSS and FBS could be due to moisture diffusion into the matrix.

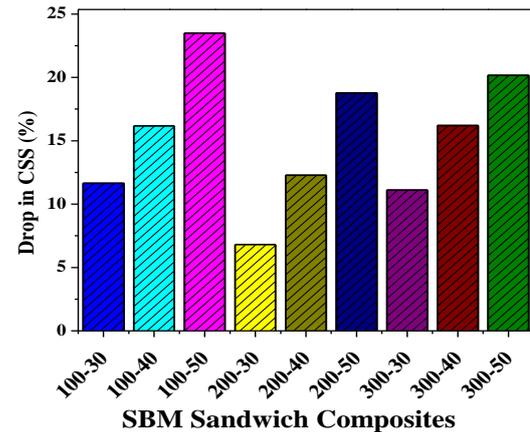
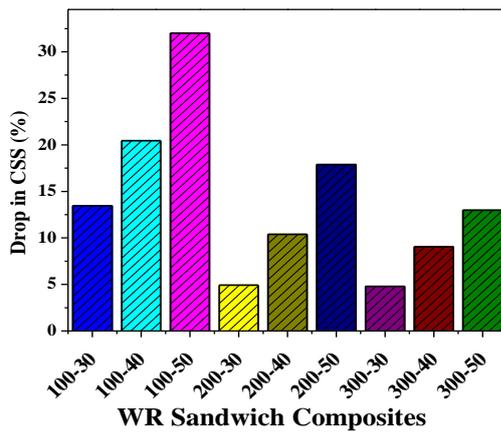
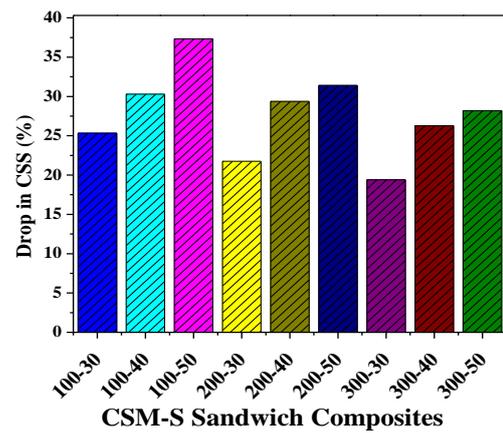
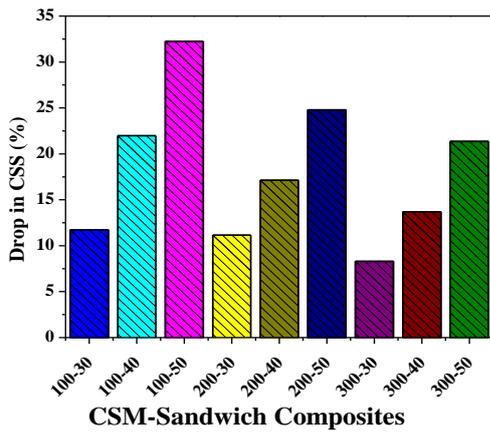


Fig. 2. Variation of Core Shear Strength of Sandwich Composites

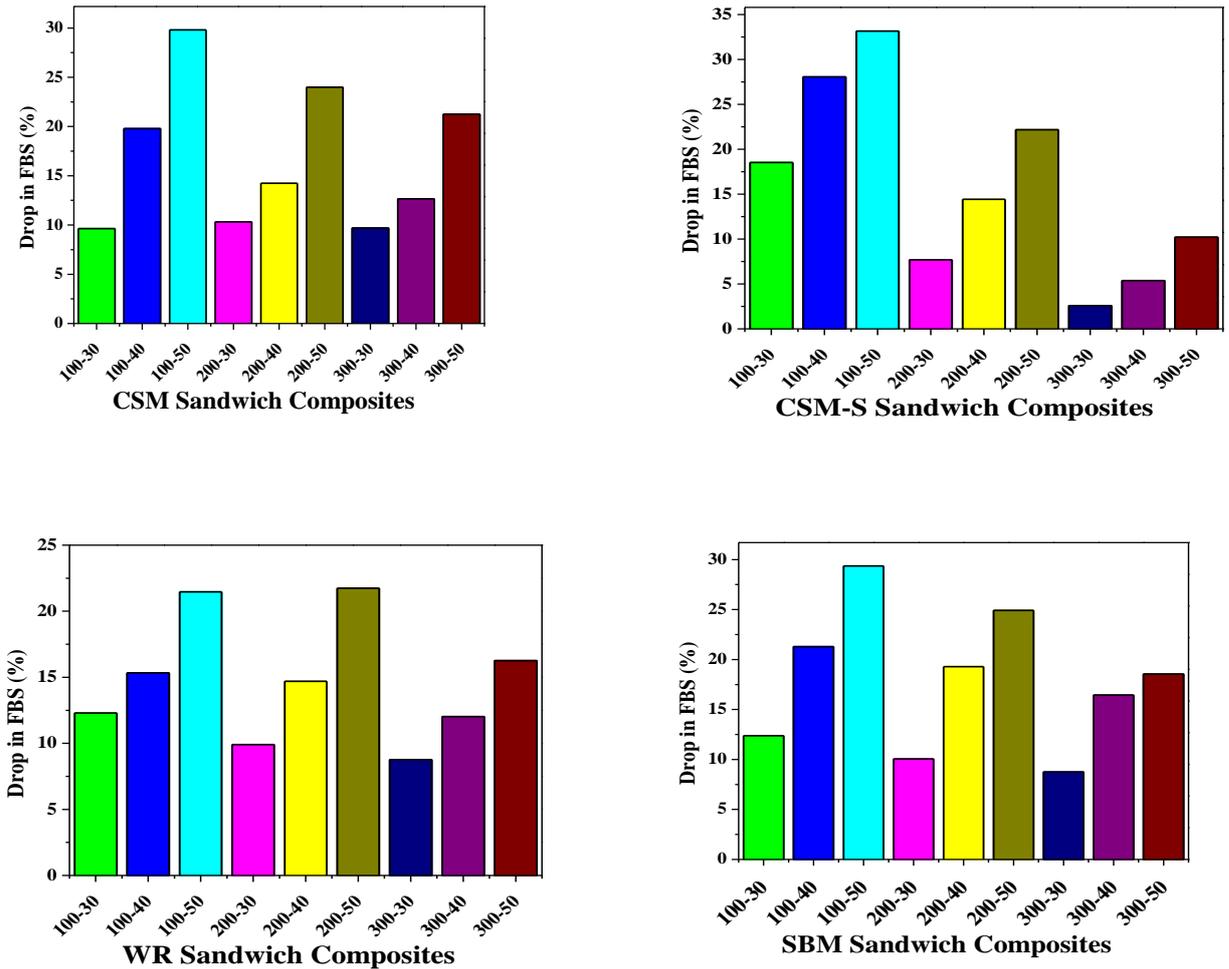
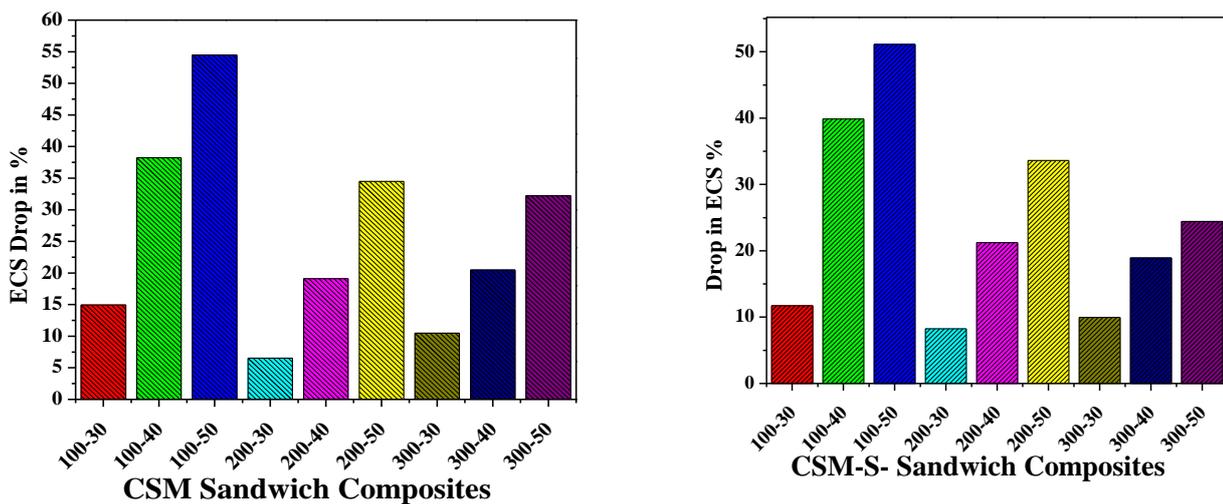


Fig. 3. Variation of Facing Bending Strength of sandwich composites

**Degradation in Compression Strength (ECS & FCS)**

All the composite specimens showed degradation in edgewise and flatwise compression strength, due to hygrothermic conditioning. The decrease in ECS values of sandwich composites as a function of PU foam core density and operating temperature are shown in Fig. 4. It is observed that the edgewise compressive strength of sandwich composites is decreased with the increase in temperature and the drop in the values of ECS are found to be more in low density composites. The percentage degradation in ECS is highest for SBM and lowest for CSM-S sandwich composites. The reason for this may be due to varying compatibility of the face sheets and PU foam core and the extent compressive load transferred to the core.



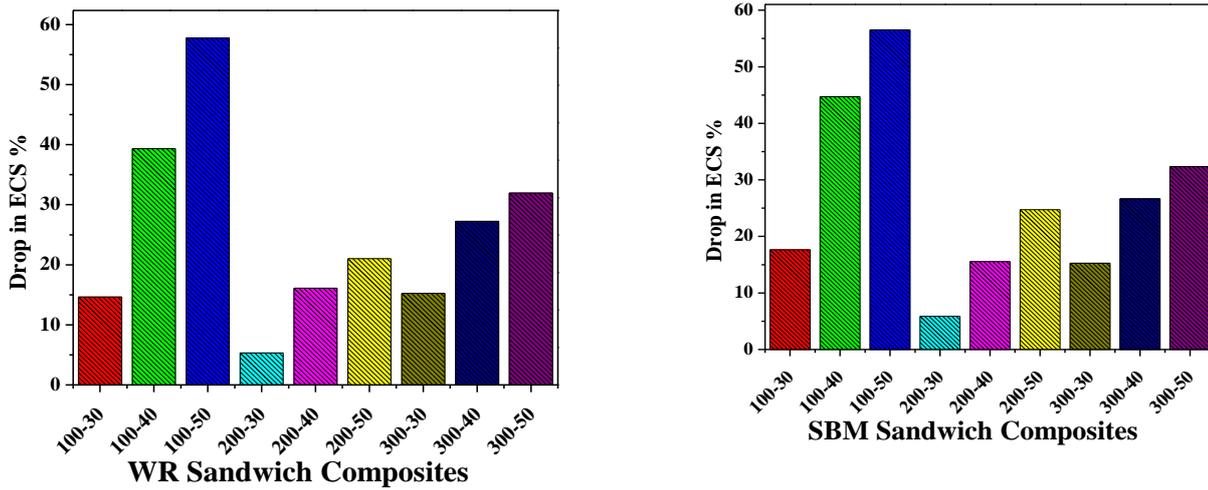


Fig. 4. Variation of Edge Compressive Strength of sandwich composites

The sandwich composites showed similar trend in degradation in the case of flatwise compression strength also. The decrease in FCS values of sandwich composites as a function of PU foam core density with operating temperature are shown in Fig. 5. It is observed that the flatwise compressive strength of sandwich composites are found decreasing with increase in temperature and the drop in the values of FCS are observed to be more in lower density composites. The percentage degradation in FCS remains almost same in all the types of the sandwich composites, as strength degradation in flatwise compressive test mode is significantly affected by the degradation of the foam core and is independent on the properties of the face sheet. The result of FCS shows that the change in fibre architecture has no significant effect on the degradation of FCS.

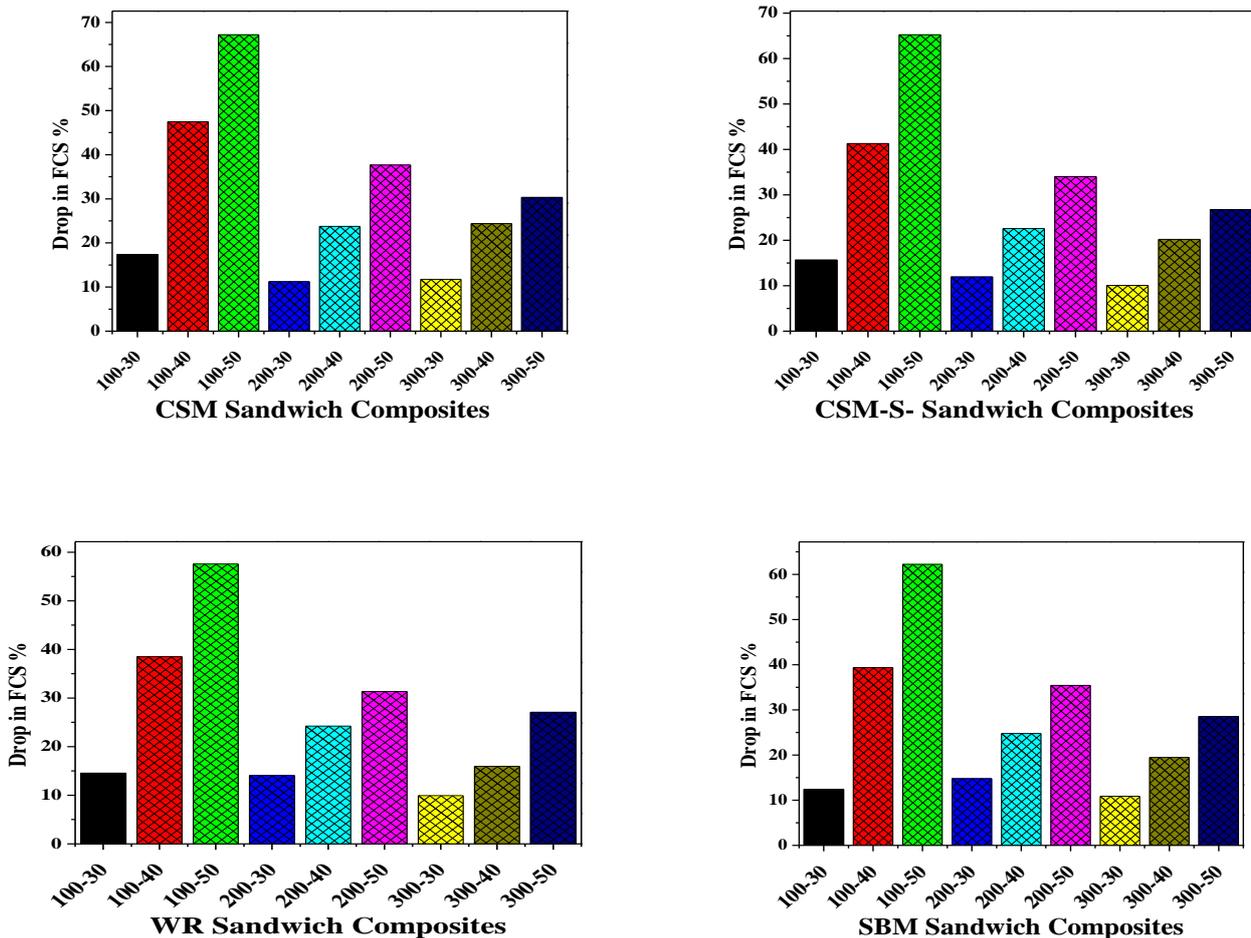


Fig. 5. Variation of Flatwise Compressive Strength of sandwich composites

### Accelerated testing for Sandwich structures

From the results obtained after exposure to salt spray for a period of four months, it is possible to predict the properties of materials for a longer period using time temperature Superposition principle (TTSP). Data collected for a period of 4 months has been used to predict the properties for a longer period. Variation of Mechanical Properties of WR- 300, CSM-300, CSM-S-300 and SBM-300 sandwich Composites are given in Table 2 to Table 5.

**Table 2. Variation of Mechanical Properties of WR -300 Composites**

Temperature	Exposure Time in months (T)	FBS (MPa)	CSS (MPa)	ECS (MPa)	FCS (MPa)
30° C	1	71.85	4.31	24.43	19.44
	2	70.86	4.26	23.72	19.06
	3	69.92	4.23	23.07	18.7
	4	67.30	4.18	22.32	18.35
40° C	1	70.85	4.22	24.04	19.23
	2	68.98	4.180	23.00	18.72
	3	66.80	4.12	22.00	18.21
	4	64.89	3.99	20.90	17.54
50° C	1	69.67	4.29	24.21	18.95
	2	67.30	4.07	23.23	18.06
	3	65.13	3.93	21.83	17.12
	4	61.77	3.82	20.87	16.04

**Table 3. Variation of Mechanical Properties of CSM -300 Composites**

Temperature	Exposure Time in months (T)	FBS (MPa)	CSS (MPa)	ECS (MPa)	FCS (MPa)
30° C	1	50.96	3.06	21.38	18.42
	2	49.62	2.99	20.92	17.98
	3	48.74	2.90	20.51	17.56
	4	46.94	2.86	20.09	17.15
40° C	1	49.62	3.01	21.02	18.03
	2	48.28	2.89	20.37	17.29
	3	46.75	2.80	19.56	16.46
	4	45.41	2.69	18.73	15.48
50° C	1	48.74	2.95	20.84	17.95
	2	46.52	2.83	19.86	17.04
	3	43.62	2.70	18.71	15.94
	4	40.94	2.57	17.45	14.81

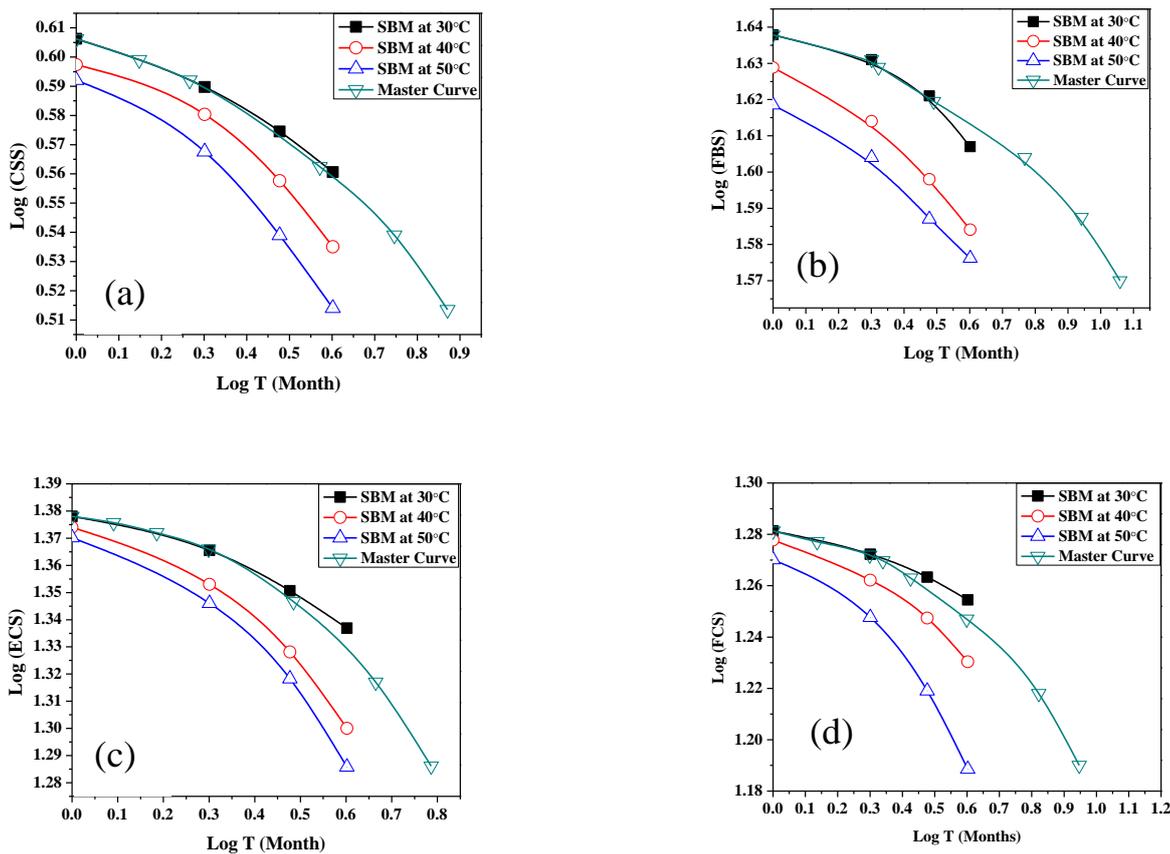
**Table 4. Variation of Mechanical Properties of CSM-S-300 Composites**

Temperature	Exposure Time in months (T)	FBS (MPa)	CSS (MPa)	ECS (MPa)	FCS (MPa)
30° C	1	64.38	3.76	21.92	18.54
	2	63.69	3.50	21.49	18.14
	3	63.24	3.27	21.09	17.75
	4	63.04	3.09	20.71	17.37
40° C	1	63.23	3.69	21.56	18.21
	2	62.58	3.43	20.77	17.52
	3	62.12	3.1	20.03	16.85
	4	61.25	2.86	19.22	16.04
50° C	1	62.59	3.61	21.38	18.03
	2	61.47	3.31	20.41	17.12
	3	60.32	2.98	19.28	16.18
	4	58.11	2.71	18.18	15.06

Log-Log plots of flexural and compression strength with time for WR-300 sandwich composites after exposing to salt spray for various temperatures is given in **Fig. 6(a-d)**. Similar types of plots are obtained for other sandwich composites also. The 30° C behavior is taken as the reference curve and the curves of 40° C and 50° C are shifted to the right until they are superimposed on the reference curve. From the master curves thus obtained, acceleration factor (k) is been calculated. Acceleration factors 'k' values are given in the **Table 6**. Acc fact increases with incesaee in dens of PU foam. The master curve obtained will give the amount of degradation as a function of time and it will be possible to find the degradation for longer duration.

**Table 5. Variation of Mechanical Properties of SBM-300 Composites**

Temperature	Exposure Time in months (T)	FBS (MPa)	CSS (MPa)	ECS (MPa)	FCS (MPa)
30° C	1	43.43	4.03	23.88	19.11
	2	42.76	3.89	23.2	18.71
	3	41.45	3.75	22.42	18.33
	4	40.33	3.63	21.72	17.96
40° C	1	42.54	3.95	23.37	18.95
	2	39.54	3.80	22.13	18.28
	3	38.43	3.61	21.01	17.67
	4	36.95	3.42	19.66	16.99
50° C	1	41.54	3.90	23.45	18.62
	2	38.54	3.69	22.18	17.68
	3	36.43	3.45	20.80	16.55
	4	36.00	3.26	19.31	15.43



**Figure 6. Log-Log plots of flexural and compression strength Vs time and Master curves for SBM-300 sandwich composites (a) CSS, (b) FBS, (c) ECS and (d) FCS**

**Table 6. Acceleration Factors 'k' values for various sandwich composites**

Sandwich Type	Acceleration Factor 'k'											
	Core Shear Strength			Facing bending Strength			Edgewise Compression Strength			Flat wise Compression Strength		
	100Kg/m <sup>3</sup>	200Kg/m <sup>3</sup>	300Kg/m <sup>3</sup>	100Kg/m <sup>3</sup>	200Kg/m <sup>3</sup>	300Kg/m <sup>3</sup>	100Kg/m <sup>3</sup>	200Kg/m <sup>3</sup>	300Kg/m <sup>3</sup>	100Kg/m <sup>3</sup>	200Kg/m <sup>3</sup>	300Kg/m <sup>3</sup>
CSM	2.5	2.45	2.39	3.5	2.74	2.47	4.02	3.56	2.21	4.86	3.06	2.19
CSM-S	1.75	1.69	1.62	2.79	2.48	2.35	3.42	3.11	2.37	4.41	3.10	2.28
WR	4.25	3.47	3.42	2.72	2.38	2.24	3.61	3.41	3.09	4.47	1.878	2.33
SBM	2.35	2.13	1.87	3.37	2.85	2.45	3.07	2.30	1.64	4.23	2.37	2.19

In the master curve obtained for WR-300 for Core Shear Strength is given in fig. 6a, it can be seen that the time ranges from log (t<sub>0</sub>) = 0 to log (t<sub>f</sub>) = 1.145. These correspond to 1 month and 13.98 months, respectively. This is 3.5 times longer than the original four months limit. Therefore the exposure to 50° C yields an acceleration factor k = 3.5 for a reference temperature of 30° C. This means that the sandwich panels exposed to salt fog and 95% RH at 50° C for a period of 1 month will have the similar degradation that of the

sandwich panels which are exposed 3.5 months at 30° C. Similarly it is possible to find the acceleration factor for other properties also. The acceleration factor for, CSS, FBS, ECS and FCS is given in Table 6. The acceleration factor depends on the fibre architecture, density of the foam and the mechanical property.

The horizontal shift required for each curve to superimpose to the master curve is the shift factor, 'a<sub>T</sub>' for that temperature. Shift factor is plotted as a function of temperature for core shear strength of WR 300 sandwich composites [Fig.7]. Similar plots are obtained for other properties also.

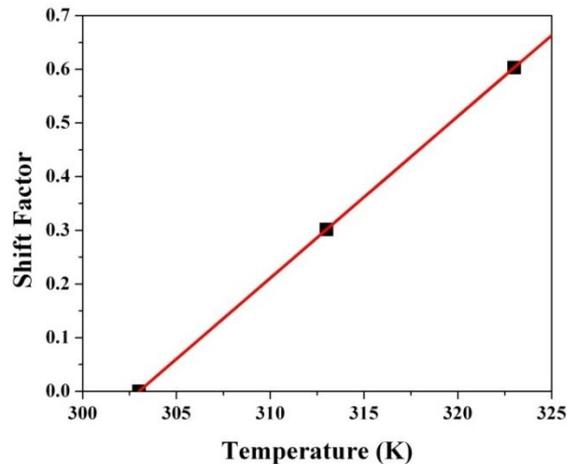


Fig 7. Shift factor V/s Temperature for WR-300 for Core Shear Strength

Arrhenius relation is used to describe the shift factor as a function of temperature. The equation obtained for CSS in the case of WR- 300 sandwich composites is given below.

$$\text{Log}a_t = 1.2279 - \frac{467.67}{T} \quad \text{----- (8)}$$

T – Temperature in (K)

This equation can be used to predict shift factors for unknown temperatures.

## Conclusion

Studies on the degradation in mechanical properties of PU foam cored E-glass reinforced sandwich composites were conducted by exposing the specimens to salt fog atmosphere at different temperatures and 95% RH. Prediction of mechanical properties of these sandwich panels under different temperatures, humidity and salt fog environment were performed by accelerated testing methodology based on the time temperature superposition principle. The three point bending (core shear strength and facing bending strength) test, edge wise and flat wise compressive tests for four different density PU foam sandwich composites were carried out.

Results reveal that the mechanical properties (CSS, FBS, ECS and FCS) of all four types of sandwich structures strongly depend on moisture and salt water absorption as well as time and temperature. Degradation in flexural and compressive properties was maximum for SBM sandwich composites and minimum for CSM-S composites. The master curves of the mechanical properties of these sandwich structures were constructed using the test data based on time temperature super position principle. From the master curves, shift factor and acceleration factor for each property have been calculated. In the present work, the master curve obtained for core shear strength of SBM-300 sandwich composites exposed at 50° C yields an acceleration factor AF=1.87 for a reference temperature of 30° C. This means that the sandwich specimens exposed to salt fog and 95% RH at 50° C for a period of 1 month will have the similar degradation as that of the sandwich composites which are exposed to 1.87 months at 30° C. This has been verified for SBM sandwich composites, the highest degrading sandwich composites out of all the four sandwich structures. The CSS strength of SBM composites at 50° C after 1 month is 3.9 MPa. It takes almost 1.9 months to degrade to 3.9 MPa at 30° C. Thus TTSP principle can be satisfactorily applied to predict long term durability. Like this we can shorten the duration of the degradation process by some acceleration factors like temperature, salt fog concentration, P<sup>H</sup> etc. and use the short-term measurements in the prediction of materials service lifetime.

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