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Fire Performance and Technological Properties of Plywood Prepared by with PMUF Adhesive Modified with Organic Phosphate.

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Abstract:

This study focuses on assessing the effectiveness of phosphate-modified phenol-melamine-urea-formaldehyde (PMUF) resin in plywood fabrication by using Eucalypatus as core veneer and gurjun sp. as face veneer. The PMUF resin synthesis was with the ratio of F:P:M:U at 4.6:2.0:0.42:0.29 by using Tricresyl phosphate at laboratory scale.12mm ply board was prepared by using the modified PMUF resin with some additive in the glue formulation. The mechanical and fire properties were carried as per respective Indian standard. FTIR, NMR, DSC-TGA analysis was carried for resin characterization for functional group confirmation. In overall from the study the data revels that the plywood made by using the phosphate modified glue shows satisfactory fire performance and mechanical properties which confirms as per IS:5509 (2000) requirement. The addition of organic phosphate enhances the plywood's fire resistance without compromising its physical and mechanical attributes, demonstrating its potential for improved safety and performance in diverse applications.

1.0 INTRODUCTION

In both residential and non-residential building construction, there has been an increase in the demand for wood and wood-based goods in the recent years. However because of their natural flammability, these goods frequently help start

Unintentional fires that cause numerous injuries and fat alities.As a result, a number of safety standards and law s relating to the spread of fire and flammability of wood prohibit its use.Timber products are frequently treated with fire retardants to enhance fire performance.Theref ore, the design of public and commercial buildings has i ncluded fireretardant treatment of plywoodand other wo od panel products for decades. However, only limited fire resistance can be achieved in wood, because no wood material is completely fire proof [Roger Pedieu et al.2012]. Because of this, the usage of wood is constrained by a number of safety standards and laws relating to its flammability and spread of fire characteristics [Laura Anne Lowden et al.2013]. The use of a solution containing urea, phosphoric acid, and ethanol as a fire retardant for wood resulted in excellent fire retardation. Flame-retardant impregnates based on alkaline silicates was used to treat wood and obtained high fire-retardant efficiency as well as high water insolubility [3].

Recycled wood waste particles were treated with a mixt ure of ammonium sulphate, diammonium phosphate, bo rax, boric acid, and ammonium bromide as a fireretarda nt agent before being utilised to make particleboards.Q

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uaternary ammonia compounds and conventional fire re tardants were used to treat plywood, which was assesse d for its fire performance and resistance to decay. [4] Heat release rates were lower for treated than untreated specimens. Time to ignite, heat release rate, extinction flammability index, thermal stability ind ex, mass loss, smoke toxicity, limiting oxygen index, su rface spread of flame, and fire resistance are the most cr ucial characteristics of flammable materials [5].Among the many polymeric materials used, epoxy resins are one of the most problematic: they are used in sectors such as; electronics or public transportation, where standards are particularly restrictive. Unfortuna-tely, they tend to burn easily while releasing high quantities of smoke and gases [6]. There are three methods by which fire-retardant plywood can be made viz. (a) coating the plywood with fire-retardant paint, (b) impregnating the plywood with fire-retardant chemicals, (c) impregnating the veneer with fire-retardant chemicals before gluing. Due to a number of different factors, production of wood composite products has risen sharply in recent years as the industry has searched for alternatives to solid wood material [9].

Products like plywood, OSB, and laminated beam mate rials are becoming more common in lightframe constru ction applicationsfor things like roof sheathing, flooring , and wall sheathing [10].Although preservatives freque ntly enhance the performance of wood composites, the practise of preservative treatment adds a new layer of c omplexity to the process of making wood composites. The biggest challenge is figuring out how to apply a tre atment method without impairing the product's structur al performance and utility before it is used [12].

Wood preservative treatments are frequently applied to composite products to improve and increase product lif e-span in order to assist combat these issues. A gene-ral review of past research conducted on this subject revealed that, for the most part, fire retardant treatments will improve the ability of a wood material to stand up to intense heat and flame for longer periods of time when compared to untreated wood. However, improvement in these extreme conditions does come at a cost to some of the other properties. Adverse effects of fire retardant treatment can include an increase in wood moisture content, increased corrosion potential, and a reduction in the strength properties of the treated material [13]. Similar research has also suggested that reductions in strength and bending properties also occur in other fire retardant treated roof sheathing products when they encounter some of the extreme temperatures that are common in roof structures [14].The basic concept is that the acidity of the chemical used, and the magnitude or the thermo-chemical exposure treated products have to endure in processing and in service, have combined to cause an effect on wood strength. Research has shown that when fire retardant treated materials are used in roofing structures, they can be cyclically exposed to extremely high temperatures for prolonged periods of time. The prolonged exposure to heat along with the high acidity of some fire retardant chemicals lead to wood products that darkened in colour, formed a dry-rot like appearance, and became very brash and brittle after a few years of service [15]. When compounded over time, those adverse effects eventually lead to product failure [16].The extensive research of the a fore-mentioned authors has lead to great advances in the knowledge about fire retardant treated wood products. Even with the extensive background research that has been conducted by these and other researchers, there are still many questions regarding the performance of these building materials. Much more research needs to be conducted to help discern why these products have a tendency to fail when they are in service [17].The main objective of this study was to evaluate the efficacy of PMUF resin modified with organic phosphate on fire performance and physic-mechanical properties of the plywood.

2.0 MATERIALS AND METHODS

2.1 Materials

Plywood was manufactured using veneers of gurjan sp. (*Diptocarpus*) of thickness 1.8 mm used as core and of thicknesses 0.3mm to 0.5 mm used as face veneers. The average moisture content after drying and before manufacturing of plywood was maintained at (5–6)%. Commercially available phenol, formaldehyde, melamine, urea, Tricresyl phosphate (TCP), and additives used for resin and glue formulation.No Hardner or catalyst was used as binder.

2.2 Methods:-

2.2.1Resin Preparation:

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Using a conventional two-
step process with a F:P:M:U molar ratio of 4.6:2:0.42:0 .29, PMUF Resin was created.

Step -1 : 200 parts of phenol and 300 parts of formaldehyde(37%) was placed in a 3 three naked pound bottom flask fitted with condenser. 24 gram of 50% sodium hydroxide solution was added. The reaction was carried for 45 minutes to 1 hour at 90 ± 2ºC.

Step -2 : 42 grams of melamine, 29 grams of urea and 162 grams of formalin was added and continued the reaction till for further 1 hour till water tolerance was 1:6/7 and flowtimein B4 cup was 15-16 seconds. Then the resin was cooled till the temperature reaches approx 60-70ºC, then Tri cresyl phosphate (TCP) was added and continue cooling till the temperature reaches to40ºC after which the resin was unloaded. The resin was kept overnight for conditioning.

2.2.2 Basic Characterization

The basic characterization of the resin is carried out industrially using inexpensive methods. The most common are:

2.2.2.1 Solid content (%) - Solid contents of the resins are determined heating 3 g of each sample in an aluminum pan, which is then placed in an oven at 120 °C, for three hours, until reaching a constant mass.

2.2.2.2 Kinematic viscosity (s) - Kinematic viscosity is determined as the resin flow time in [s] using a flow cup viscometer. The viscocity of the resin was determined in terms of flow time in B4 cup.

2.2.2.3 Density (g/cm3) - It is usually determined based on the weight/volume ratio, and measured using a hydrometer.

2.2.2.4 Gel Time (s) - The gel time was measured in three replicate in gel time meter @100^{0}C.

2.2.2.5 pH-value - The pH is monitored automatically in a pH meter.

2.2.2.6Water tolerance - The water tolerance of the resins is evaluated by transferring 5 mL of resin (at 25 °C) to a test tube, and determining the needed amount of distilled water to be added for it to become cloudy. The water tolerance is given according to the following expression:

Water Tolerance = V' : V

where V is the distilled water added (mL), and V' is total sample volume (mL).

2.2.2.7Free formaldehyde – The determination of free formaldehyde in amino resins is accomplished according to the European Standard EN 1243. Accordingly, this test is based on the reaction of the free formaldehyde with sodium sulphite in the presence of a measured excess of acid, in the following stoichiometry:

$CH2O + Na2SO3 + H2O \rightarrow CH2(OH)SO3Na + NaOH$

The acid-sulphite mixture provides an essentially neutral buffered system, which prevents hydrolysis of condensed formaldehyde. A reaction temperature close to 0 °C helps to ensure the absence of side reactions. In the end, the alkaline titration of the unreacted excess acid is performed.

The free formaldehyde content shall be calculated by the following formula:

2.2.2.8 Free formaldeyhde %= $(\boldsymbol{V2}-\boldsymbol{V1}) \times \boldsymbol{M} \times \boldsymbol{3.002m}$

where: $V1$ is the volume (mL) of 0.1 M sodium hydroxide solution used for the adhesive test; $V2$ is the volume (mL) of 0.1 M sodium hydroxide solution used for the blank test; M is the molarity of sodium hydroxide solution; m is the mass (g) of the adhesive test portion.

2.2.3 Spectroscopic and Thermal Analysis

2.2.3.1Differential scanning calorimetry (DSC) measures the change of the difference in the heat flow rate between a sample and the reference sample, while they are subjected to a controlled temperature program. The employment of such technique has been useful to better understanding of the thermal curing process of melamine-formaldehyde resins, namely the existence of two steps of reaction. The differential scanning calorimeter (DSC 204 F1) produced by Nez was used for the test. The test parameters were as follows: temperature 25~210 °C; heating rate 10 °C/min; N_2 protection; sample mass 5–8 mg

2.2.3.2Thermogravimetric analysis (TGA) determines the change in weight as a function of a temperature program under a controlled atmosphere. The weight loss of the sample is recorded as a function of the temperature, which is related to the respective volatiles content. The thermal decomposition of PMUF resins can be studied by this technique.

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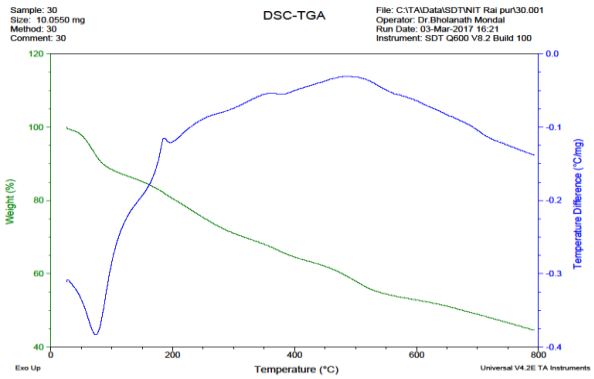
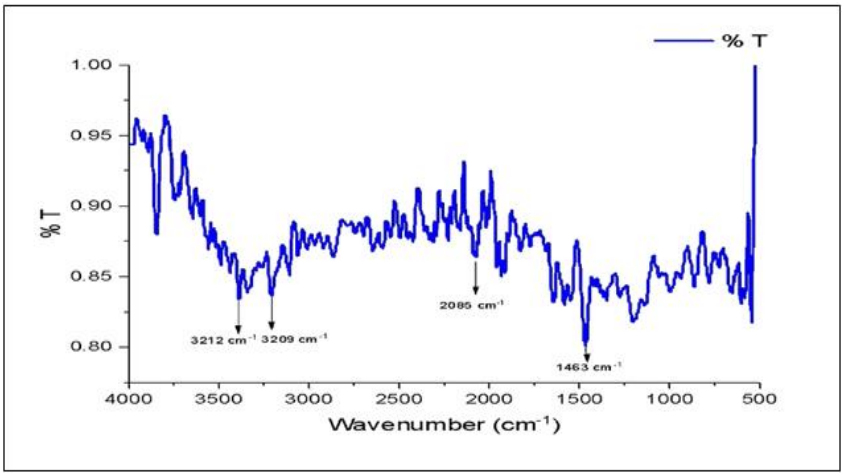


Fig 1: DSC & TGA – Therrmal behaviour study of the adhesive

2.2.3.3Test of Fourier transform infrared Analysis (FTIR) :-

Functional group analysis of liquid adhesive was performed using Fourier transform infrared (FTIR Spectrum Two, Perkin Elmer, Waltham, MA, USA) using the universal attenuated total reflectance (UATR) method. The samples were placed in a sample holder, pressed, and scanned from 400 to 4000 cm^{-1} with an average of 16 scans at a resolution of 4 cm^{-1}.



Test of ^{13}C Nuclear Magnetic Resonance (^{13}C–NMR)

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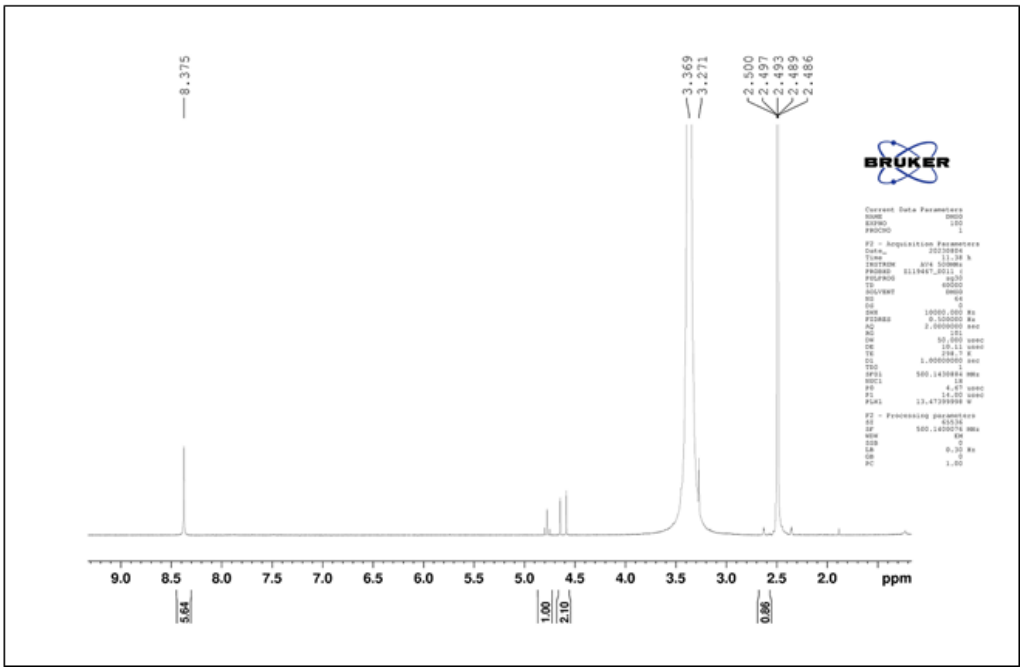
2.2.3.4(NMR) spectroscopy is an useful technique to identify and analyse organic compounds [49]. 13C NMR has been used to investigate melamine resins Quantities of 100 µL of LMUF resin and 300 µL of DMSO–d_6 were injected into a nuclear magnetic tube, dissolved and shaken, and tested by a Bruker Avance high–resolution superconducting over frequency nuclear magnetic resonance instrument. The test parameters were as follows: pulse sequence, zgig; internal standard, DMSO–d_6; cumulative times, 500–800 times; spectral width, 39,062.5 Hz



2.2.4 Adhesive mix Formulation

The adhesive mixture was created by combining synthe tic resin, additives, and different concentrations of exte nsion level resin while stirring with a speed-controlled stirrer.

Until a homogenous mixture was formed, the mixing w as maintained for another 30 minutes. Viscosity of the glue after mixing was taken in B_6 Cup (IS:3944-1982).The optimized glue formulation was given in

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Table 1: Adhesive formulation

Sl.no	Resin and additives	Parts
1	PMUF	100
2	Precipitate silica	04
3	Wheat flour	05
4	Di-sodium octra borate tetra hydrate	02

2.2.5 Preparation of Specimen

Two sets of 12mm thick plywood were manufactured by using Gurjan sp. (*Dipterocarpus*) species as core, face veneer of size 2ft x 2ft. Adhesive mix was applied to each core veneer by brushing and conditioned for moisture content up to 14% at a spread rat 280 - 300gms/m^2 on both the sides. Then the assembled veneer was pressed in a hydraulic hot press @130 - 135^{0}C 11-12 Kg/Cm2 Sp. Pressure for 15 minutes for 12mm plywood.

Before preparing the samples for testing in accordance with IS:5509 (2000) and IS 1734 –Part-3 (1983), the plywood specimen was conditioned for 7 days to establi sh equilibrium mixture at 23^{o}C and 50% RH. The samples were marked as “A”, “B”, and “C” according to plywood constructed with conventional resin, PMUF resin and PMUF resin with TCP and additive whose details are given below:

Sample A: Control fire retardant plywood with PF resin used as conventionally

Sample B: Fire retardant plywood with PMUF resin

Sample C: Fire retardant plywood with PMUF with TCP and additive.

2.2.6 Fire Retardancy Test

Coated and uncoated specimens of special dimensions were prepared and subjected to different tests. To assess the fire retardancy properties, the following tests were be carried out:

(i) Flame penetration test as per IS : 1734- Part-3 (1983)
(ii) Flammability test as per IS : 1734- Part-3 (1983)
(iii) Rate of Burning test as per IS : 1734- Part-3 (1983)

The plywood samples manufactured with PMUF adhesive and adhesive with additive were tested conforming to IS: 5590 (2000). To assess the fire retardancy properties of the plywood flammability test, flame penetration test and rate of burning test were carried out.

2.2.6.1 Flammability test

Flammability is a general word used to describe the reaction to fire behavior of a material and the test has been carried out as per IS: 1734- Part-3 (1983). Each test specimen was created with dimensions of 125 mm x 125 mm and full material thickness. The specimen were preconditioned to a constant mass at a relative humidity of 65 ± 5% and at a temperature of 27 ± 2°C.The test specimens were held vertical 15 mm apart, one specimen being held 40 mm higher than the other. An ordinary Bunsen burner having 3 mm bore was fixed horizontally so that the flame plays against the lower end of the inner face of the lower specimen (Fig 4).

The burner's end was 12 mm away from the specimen's face, with the burner's axis located 22 mm above the lo wer edge of the lower specimen.LPG was fed to the burner resulting in a blue flame which when unobstructed was 50 mm long. The flame ignites the lower specimen's face, which then ignites the higher specimen's opposing face. After the lower specimen ignited; the amount of time it took for the higher s pecimen to ignite was noted. This ignition was usually very distinct and capable of being timed to a few seconds. (Ref Table 5)

2.2.6.2 Flame penetration test

Flame penetration test was carried out as 1734- Part-3 (1983). Each test specimen were prepared of size having full thickness of the material and approximately 125mm ×125 mm. The test specimen was held horizontally 50 mm above the nozzle of a blow-pipe flame (Fig 4). The test specimen was then rotated in a horizontal plane at 75 rev/min in such a way that the centre of the flame describes a circle of 25 mm diameter. The amount of time it took for the flame to pierce the plywood thickness was noted. (Ref Table 5)

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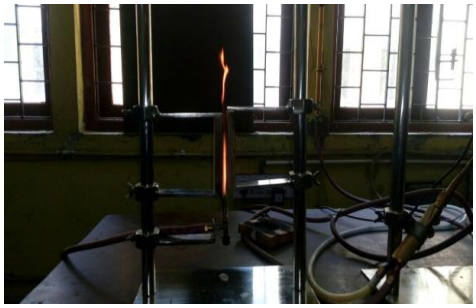

Fig 2 Apparatus for flammability test

Fig 3 Apparatus for rate of burning test

Fig 4 Apparatus for Flame penetration test

2.2.6.3 Rate of burning test

Testing for burning rate was done in accordance with I S: 1734-Part-3 (1983).

Each test specimen was made to measure, measuring ro ughly 100 mm by 12.5 mm and including the entire thic kness of the material.The samples were preconditioned to a constant mass at a temperature of 27 2 °C and a rel ative humidity of 65 5%.The test object was suspended in a fire tube and set to hang 30 mm above the burner's flame (Fig. 3).

A blue flame was then used to burn the test specimen, a nd the time it took for each 10% loss in mass was noted .

The duration between a 30% and 70% reduction in mas s was timed and recorded for comparison.

(Ref. Table 5)

2.2.7 Study of Mechanical Properties

Physico- mechanical properties of the fire retardant plywood has been carried as per IS: 1734 viz Modulus of Rupture, Modulus of Elasticity, Glue shear strength in 20 Ton capacity Universal testing machine.

2.2.8 Water resistance test

In order to assess the water resistance of the plywood, cyclic test was carried as per IS: 848 (2006).The Plywood samples were exposed in boiling water for 8 hrs. And then dried @65±2^{0}C for 16Hrs. the same process was continued for three cycles.

3.0 RESULT AND DISCUSSION

3.1 Adhesive characterization:

Recent modification of PMUF resin was aimed at increasing fire retardancy properties as well as bonding strength. A wide spectrum of phosphate compound was applied as modifying agent to increase the fire retardancy properties of the board, however it was observed that using different phosphate compounds in PMUF resin modification enhances the physical properties and fire retardancy properties (Ref Table 5) but dominates bonding strength after incorporation with resin. Solid content was about 48% on average and was not affected much by the insertion of phosphate modifier like TCP. In addition density and viscosity remained unchanged. The fig 1 indicates gel time changes w.r.t % incorporation of TCP to PMU resin.

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Table 2-PMUF Resin Properties :-

Properties	PMUF resin	PMUF resin with TCP and additive
Solid content (%)	48.6	48.8
Free formaldehyde (%)	0.46	0.42
Density(g/cm^3)	1.206	1.195
Viscosity(CSP)	182	187
Gel time(S)	55	62

3.2 Effect of Technological Properties of the plywood

The use of PMUF adhesive modified with organic phosphate in the preparation of plywood have a significant effect on the mechanical properties of the plywood .The MOR and MOE values meets the required specification as per IS :5509 (2000) (Ref Table 5). .The tensile strength of the plywood sample shows an improvement then the plywood made with traditional adhesive. The presence of organic phosphate likely contributes to better interfacial adhesion between wood veneers, leading to increased tensile strength. Shear strength is essential for the structural integrity of plywood, especially in applications where it will be subjected to lateral forces. Shear strength is essential for the structural integrity of plywood, especially in applications where it will be subjected to lateral forces. From the results it has been shown that shear strength of the plywood sample gave satisfactory results which meets as per the requirement of Indian standards. From the cyclic test the sample passed three cycles in boiling water which shows the adhesive bonding has significant water resistance power. The modified PMUF adhesive with organic phosphate shows not only enhanced fire performance ,it shows also improved mechanical properties making it suitable for a wider range of applications where these properties are crucial.

Table 3: Glue shear strength of 12mm thick plywood made with modified PMUF resin

Properties	PMUF resin	PMUF resin with TCP
GSS(N)	1480	1320
Tensile strength(N/mm^2)	93	86

Table 4: Resistance to water test of the 12mm thick plywood

Test	Test Method	Criteria for conformity	Results
Three cycles : Each cycle consisting of 8 hours boiling in water and thereafter drying at 65±20C for 16 hours.	Clause 4 and 7.3.2 of IS:848 -2006(as per table -1)	No separation of plies at the edges and /or surface at the end of three cycles. On forcible separation of plies with knife, wood failure shall be predominant and shall be more than 75% for excellent bond and not less than 50% for pass standard. For less than 50% wood failure, the specimen shall be considered as failed.	No separation of plies at the edges and /or surface at the end of three cycles. 60% Wood Failure Pass Standard

Table 5: Physico-mechanical properties of fire retardant plywood

Sample	Glue Shear Strength				Static Bending Strength			
	Dry State		Wet State (BWR Grade)		MoR (N/mm^2)		MoE (N/mm^2)	
	Load (N)	Wood Failure (%)	Load (N)	Wood Failure (%)	Along the grain	Across the grin	Along the grain	Across the grain
A	1410	70	1020	60	52.48	42.45	5510	3367
B	1420	75	1060	65	50.64	41.75	5482	3486
C	1340	80	1010	65	48.68	41.62	5086	3246

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Requirement as per BWP grade plywood	1350	60	1000	60	--	--	--	--
Requirement as per BWR grade plywood	--	--	--	--	40.00	20.00	5000	2500

3.3 Effect of fire retardant chemicals on incombustible properties:

It has been shown that the substrate decreases heat pene tration in the presence of heat or flame, slowing the pro pagation of the flame and postponing structural failure. The results of fire retardancy complies with the requirement of IS: 5509 (2000) (Ref Table 6). The adhesive and additive composition significantly improved the fire retardancy and bonding qualities of the veneer by impregnating it with chemicals, making the wood more rigid and brittle. Under the heating action, phosphorous compounds decomposes at lower temperature to give phosphoric acid then creates an in tumescent protection layer which prevents further oxidation and improves the char formation. Boron compounds break down under heat to create a glossy protective layer that serves as a barrier for the oxidation of polymer chains. Both phosphor-rous and boron compounds also act as smoke suppressant. Silicate compounds act as high thermal stability. The fire retardant plywood manufactured with the developed adhesive and additive shows a significant fire retardant properties tested as per IS:5509 (2000) (Ref Table 6).The results from the mechanical tests shows that the new fire retardant combination exhibits superior mechanical strength.

Table 5 : Fire Retardancy properties of 12 mm thick plywood tested as per IS 5509:2000

Description of sample	Flame penetration (minutes)	Flammability (minutes)	Rate of burning (minutes)
Sample A	28	31	12
Sample B	42	40	15
Sample C	48	51	21
Requirement as per IS5509:2000	30	30	20

3.4 Structural and Thermal Analysis:-

The stretching vibrations in the IR spectrum often correspond to the movement of chemical bonds. The value of 3212 cm^{-1} mentioned in IR spectra is typically associated with the stretching vibration of the hydroxyl (OH) group, specifically in alcohols (R-OH) or phenols (Ar-OH). The stretching value at 1463 cm^{-1} in an infrared (IR) spectrum is often associated with the presence of a methylene group (CH2) in a molecule. This absorption band is typical for the symmetric bending vibration of the CH2 group. Methylene groups are commonly found in various organic compounds, including alkanes, alkenes, and alkynes.

In nuclear magnetic resonance (NMR) spectroscopy, the peaks in the spectrum correspond to different types of nuclei within a molecule and provide information about their chemical environment. The peak at 3.2 in a proton (^1H) NMR spectrum typically represents the protons (hydrogen nuclei) in a compound that are adjacent to an electronegative atom, often an oxygen (O) or a nitrogen (N) atom. In proton (^1H) NMR spectroscopy, a peak at around 2.4 ppm (parts per million) is often associated with protons on a methyl (CH_3) group. This region is commonly referred to as the "methyl" region of the NMR spectrum. Methyl groups are frequently found in organic compounds and have distinctive chemical shifts in the NMR spectrum.

The curing degree is an important index to measure the curing reaction of adhesives. The curing degree curve of PMUF resins obtained by integrating the DSC peaks, there was a relatively low loss of mass in this stage. 150–400 °C this was a stage of thermal decomposition of the major skeleton of the PMUF resins, involving the breakage and decomposition of ether bonds, the release

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of formaldehyde, and the breakage and decomposition of polyethylene bonds

4.0 CONCLUSION

The study aimed to assess both the fire performance and technological properties of plywood manufactured using this adhesive modification the incorporation of organic phosphate into PMUF adhesive has shown promise in enhancing the fire resistance of plywood The Synergetic effect of phosphate with boron compound enhances the fire performance of the ply board. Hence the plywood made with phosphate modified PMUF adhesive and with additives like silica, Disodium octaborate tetra hydrate (DOT) shows good performance in fire retardancy as well as Physico-mechanical properties like bonding strength, Mechanical strength and water resistance properties. The results achieved from this work confirms as per the requirement of IS :5509 (2000) .In summary, the modification of PMUF adhesive with organic phosphate has shown potential for enhancing the fire performance and technological properties of plywood. This innovation holds promise for improving the safety and durability of plywood-based products, particularly in construction and other applications where fire resistance and mechanical strength are critical factors.

5.0ACKNOWLEDGEMENT

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