# BLACK FIGURE ASELVA OF RESIVE FOR FIRE RETARDANT.

In this paper S/Shri N.K. Sexena & Suril K. Sharma, Scientists, Fire Research Laboratory, Central Building Research Institute, Roorkee have based on the various experiments carried out by them, brought out that black icour can be effectively used to replace costly resins used as binders in the manufacture of wood based panel products commonly used in buildings. The resins presently used are very costly while black liquor which is a by product of paper industry is presently posing disposal and environmental pollution problems.

Editor

## INTRODUCTION

The wood based panel products like plywood, particle board, fibre board etc. are generally used in buildings for various purposes. These materials are manufactured by using wood and agricultural wastes by bonding with a suitable binder under the controlled conditions of temperature, pressure and catalyst. Two major problems are noted in using these materials i.e. combustible nature of these products and the cost of adhesives. Synthetic resins like urea formaldehyde (U.F.) and phenol formaldehyde (P.F.) are generally used for the manufacture of various panel products. The cost of these resins is very high in India, due to the scarce availability of ingredients like urea, phenol, formalin etc. Various natural phenolic substances like tanin, lignin, tar acid, C.N.S.L. etc. have been used to substitute phenol for the preparation of phenol-formaldehyde resin for panel products1-5

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Lignin is a natural binding material in all plants. The basic structural unit of lignin is a phenyl propane unit and it contains methoxyl and hydroxyl groups, part of which are phenolic in nature structure of these lignins varies from species to species<sup>7,8</sup>.

One major source of lignin is the black liquor, a waste by product obtained during pulping of various fibrous raw materials in paper mills. At present most paper mills are burning this black liquor for recovery of the pulping chemicals which may not be the proper utilization of this source of natural phenolic substance. Thus utilization of black liquor to substitute phenol in the preparation of phenol formaldehyde resin for panel products offers great potential due to large availability from paper mills.

The present communication deals with the development of adhesive from black liquor replacing phenol which has been utilized in making fire retardant particle board.

#### **EXPERIMENTAL**

#### Materials and Methods

All the reagents used in this investigation

were of L.R. grade. Rice husk and wood chips were procured from the local market.

## Preparation of black liquor

Black liquor was prepared by pulping rice husk in an autoclave at appropriate condition of temperature and pressure. The conditions were as:

Alkali 12-14 %
Bath Ratio 1:6
Temperature 165  $\pm$  5°C
Time 4 hrs
Pressure 1.8 kg/cm<sup>2</sup>

Black liquor was collected after filtering and then kept in air tight container.

## **Analysis of Black Liquor**

Black liquor obtained from rice husk was analysed for various physico-chemical properties following standard methods. The results are recorded in Table-1.

#### Preparation of Resin from Black Liquor

Black liquor phenol-formaldehyde (BLPF) resins were formulated, replacing phenol by black liquor of rice husk. For comparing purposes a phenol formaldehyde (PF) resin formulated only with phenol and formaldehyde was also prepared.

Resins were prepared by reacting phenol with 37% formalin (1:1.8,w/w) ratio in the presence of an alkaline catalyst. Phenol was replaced with varying amount of black liquor. During preparation, ph was adjusted to 9.5 in a reactor for 40-50 minutes at 90°C. The resin so prepared was allowed to cool down and stored in an air tight container. Solid content and density of different resins were determined. Formulation and characteristics of resins are given in Table-2.

## Preparation of Particle Board

To prepare particle board dried wood chips were taken and 9-10 percent of BLPF resin was sprayed on them and mixed properly. The resin blended particles were formed into a mat in a mould. The formed particle mat was pressed in a hot press at 180-220°C and 18-22 Kg/cm²

pressure for 20-40 minmutes and then taken out. A few particle boards were also prepared using PF resin.

To achieve fire retardancy in particle boards a few boards were also prepared using fire retardant chemicals based on phosphates and amides (1:2.2,w/w) ratio and 15-18 percent of wood chips. Boards were conditioned at 65 percent R.H. and 27± 2°C for one week and then evaluated for their physical and mechanical properties.

#### **Evaluation of Performance of the Product**

A few characteristics such as density, moisture content, modulus of rupture (M.O.R.) and water absorption were evaluated following Indian Standards IS: 2380 and IS: 3087 methods<sup>9,10</sup>. Effect of fire retardants on M.O.R. and moisture absorption was also determined. Data is recorded in Table — 3.

Fire performance was evaluated by exposing the specimens to severe conditions. For exposure, a Bunsen burner of 9 mm inner dia with LPG as a fuel was used. Specimen of particle board (8x150x305mm) were hanged vertically with the help of hooks. The gas burner of about 50 mm flame height was placed in such a way that the base of the flame was 25 mm below the middle of the lower edge of the specimen. The flame was applied for different time upto 30 minutes. After that, it was withdrawn and then after flame time, after glow time and weight loss were measured. Results are given in Table —4.

#### **RESULTS AND DISCUSSION**

The analysis of black liquor from rice husk showed that the solid contents were 14.86 percent consisting of dissolved and suspended solids. Liquin was found 10.92 percent in the rice husk black liquor. The pH 9.5-10 of the black liquor shows that it was having unused alkali. Table — 2 shows the characteristics of adhesives in which phenol was replaced with black liquor in varying proportions. It was observed that as the amount of black liquor increased in the resin preparation, the solid contents of the resin goes on decreasing. It was experimentally established that as the solid content of the resins decreases, higher temperature and longer duration are required to obtain maximum strength of the product.

It is observed from the data of modulus of rupture that the product developed with upto 60 percent replacement of phenol with black liquor adhesive meet the requirement of IS: 2380 and IS: 3087. At this, replacement, product did not pass the requirement specified in the standard for water

absorption. It is also observed that addition of fire retardants increase the modulus of rupture and decrease the water absorption characteristics of the particle board. The product developed with the addition of fire retardants meet the requirement of water absorption even with 60 percent replacement of phenol (Table - 3).

From burning test data it is noted that combination of phosphate and amide studied, is found quite effective in reducing the burning characteristics of the particle boards. The specimens of particle boards were burnt completely within 1.5 minutes with flame and afterglow combustion while specimens having fire retardants did not ignite under the similar conditions of exposure (Table — 4).

#### CONCLUSION

Wood based panel products are very much used in buildings for diverse applications. These materials are highly combustible and pose great fire hazard. The resins used for making these products are also very costly in India. Black liquor which is a by product of paper industry presently posing disposal and environmental pollution problems can profitably be utilized to obtain cheaper resins. It is concluded that materials developed using black liquor based adhesive satisfied the requirements specified in the standards. The fire retardants studied were found effective in reducing the burning characterisitics of the product. It is also concluded that fire retardants are also effective in increasing the modulus of rupture and decreasing water absorption characteristics of the product. Thus black liquor based adhesives may be utilized in developing cheaper materials having improved fire performance and physical characteristics.

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# TABLE - 1

## PROPERTIES OF BLCK LIQUOR

Solid Contents

14.86 %

Density at 20<sup>o</sup>C

1.06 gm/ml

Lignin Concentration 10.9

10.92 %

 $\mathsf{P}^\mathsf{h}$ 

9.5-1.0

## TABLE - 2

# FORMULATIONS AND CHARACTERISTICS OF BLPF RESINS

SI. No.	Phenoi Replaced (%)	Black Liquor (gm)	Phenol (gms)	Formalin (gms)	Density gm/cc	Solid Content (%)
4		. '			1.10	49.6
1.	10	20	180	360	i i	
<b>2</b> .	20	30	120	270	1.10	46.7
3.	30	54	126	324	1.08	43.1
4.	40	126	189	567	1.07	41.9
5.	50	170	170	612	1.05	36.7
6.	60	180	120	540	1.05	33.4
7.	70	210	90	540	1.05	29.2
8.	80	160	40	360	1.03	23.8
9.	90	135	15	270	1.02	17.2
10.	100	175	00	315	1.02	13.5
11.	00	00	150	270	1.08	51.6

## TABLE - 3

## CHARACTERISTICS OF PARTICLE BOARD

Resin Used	Type of Board	Density (Kg/m³)	Water Absorption (%) After 24 Hrs.	M.O.R. (Kg/cm <sup>2</sup> )
PF	Flat Pressed Single Layer (FPS)	660-832	050.50	162.00
BLPF-20	-do-	-do-	048.60	170.00
BLPF-40	-do-	-do-	042.80	170.00
BLPF-60	-do-	-do-	074.50	134.20
BLPF-80	-do-	-do-	112.70	032.00
BLPF-100	-do-	-do-	131.20	003.90
PF+FR Additives (FRA)	-do-	-do-	046.00	164.00
BLPF-20+FRA	-do-	-do-	043.50	167.20
BLPF-40+FRA	<b>-d</b> o-	<b>-d</b> o-	037.75	193.60
BLPF-60+FRA	-do-	-do-	067.90	171.70
BLPF-80+FRA	-do-	-do-	103.20	041.20
BLPF-100+FRA	<b>-d</b> o-	<b>-d</b> 0-	129.50	005.20

BLPF-20, 40 .....phenol replaced.

TABLE - 4
FIRE PERFORMANCE OF THE BOARD

Sample	intial Wt. of Specimen (gms)	Time of exposure (min.)	After flame Time (sec)	After glow Time (sec)	Wt. loss (gms)	Remark
PF+FRA	54.8	05	00	000	0.69	Self extinguished
-do-	55.4	10	00	000	3.20	-do-
-do-	54.5	30	06	600	12.40	After glow combustion observed after that self extinguished.
BLPF-40	55.2	05	00	000	0.52	Self extinguished
+FRA	54.7	10	00	000	2.40	-do-
j	55.6	30	04	282	9.80	After glow observed after that self extinguished
PF	55.2	05	82	Cont.	Burnt	Completely burnt with after glow combustion within 1.5 minutes.
BLPF	54.7	05	67	Cont.	Burnt	-do-