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A STEP TOWARDS PLYWOOD PRESERVATION THROUGH BORIC AND SILICIC ACID COMBINATION

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ABSTRACT
Poplar is one of the most economic agro crop, shown its potential to fulfill the demand of raw material in plywood market. Although, plywood manufactured from poplar being a non durable timber species is more susceptible to biological attack and needs preservative treatment in service. Therefore, the present work was done with object to develop a new eco safe preservative combination for plywood industries. The study was conducted to evaluate the compatibility of boric acid, silicic acid and their combinations with phenol formaldehyde adhesive in plywood made of *Populus deltoides*. Glue shear strength was tested to evaluate compatibility of the different compositions with adhesive. The efficacy of these chemicals was also determined against wood decaying fungi through soil block bioassay. Results exhibited that boric acid is effective preservative, but not imparted satisfactory shear strength in dry and wet state. While, silicic acid alone is not effective as preservative, but it performed satisfactory strength criteria laid down by IS: 848 (1974). Plywood treated with combinations of silicic and boric acid was found effective against test fungi (brown and white rot) and also performed well in compatibility test.

KEY WORDS: Compatibility, glue shear strength, mycological test, weight loss**.**

INTRODUCTION

Plywood, fiberboard and particle board are three important wood based composite panels in India. A major portion of Indian wood based market is covered by plywood *i.e*. 78% and rest is covered by medium density fiber board and particle board. The annual market of plywood and particle board was reported worth of Rs. 17 billion in India (Capexil, 2009). Oak, cherry, poplar, maple, larch, fir, pines *etc*. are commonly used timber species for plywood manufacturing (Anon, 2002). Among these poplar (*Populus deltoides Bartr Ex. Marsh*) attracted wood industries most due to its easy availability and suitability for plywood manufacturing. It is fast growing timber species and has shown potential to generate high economic value for farmers (Tewari, 1993). Plywood and the match industries is the biggest consumer of poplar (Bansal *et al.*, 1999). According to Kishwan and Kumar (2003), 82% of poplar goes to plywood industries in Yamunanagar market of Haryana (India). It is expected that demand of poplar plywood will increase up to 38 MM^3 till 2020 in India (Singh, 2012). Simultaneously, the use of plywood in moisture-prone applications is increased, which subject them to conditions suitable for mold, stain and decay (Fogel and Lloyd, 2002). Therefore it is desirable to improve life span of non durable species through some preservative treatment, since annual replacement cost of untreated wood in service is much higher than treated wood. Preservative treatment of solid wood is established and easy as compared to composite wood, since treated composite panels are difficult to bond (Vick *et al.,* 1990). The problem associated with the use of preservative in plywood is its compatibility with adhesive used. Wood preservatives being a chemical may interfere with the adhesive and manufacturing conditions which may lead the poor bonding (Barnes and Amburgey, 1993). Sometimes preservative may reduce the wettability of the wood surface or physically block, which prevents the bonding of adhesive to wood. Therefore, compatibility of preservatives with adhesive in different composite panels has to be considered separately, because it may result the product of low strength.

Boric acid is well known preservative and fire retardants and has low mammalian toxicity (Yalinkinlic *et al*., 1999). But its leachable nature limits its use in field conditions. Many efforts have been done in this direction such as use of zinc borates (Kirkpatrick and Barnes, 2006), organic esters (Humphrey *et al.,* 2002), silicone gels (Yamaguchi, 2001), animal proteins (Mazela *et al.,* 2007) *etc*. Boron was successfully combined with silicic acid in lattice structure of wood by Yamaguchi (2002, 2003 and 2005). Besides good fixation of boron, the efficacy of the combination was recorded against fungi and termite in solid wood (Yamaguchi, 2005). The application of boric and silicic acid in combination was studied in only solid wood, not in plywood so far. It can also be one of the alternate of the conventional preservatives and give a new direction to plywood treatment. Therefore, the objective of the present study is to evaluate the efficacy and compatibility of different concentrations of boric acid, silicic acid and their combinations with phenol formaldehyde adhesive in plywood in direction to develop a new eco friendly preservative combination for composites.

MATERIALS & METHODS

Plywood manufacturing

The plywood was prepared from *Populus deltoides* Bartr Ex. Marsh in Forest Research Institute Dehradun (IS: 303, 1989). The logs of green poplar of average length of 125 cm and girth of 150 cm were procured from Forest Research Institute Dehradun (Latitude $28^0$43' to $31^0$27' N and Longitude $77^0$34' to $81^0$02' E) and peeled off to veneers of 0.16 cm thickness. After peeling the veneers were visually graded and clipped into required sizes for plywood preparation. The clipped veneers were dried to 8 ±2% moisture content prior to treatment. After that resol type Phenol formaldehyde adhesive (PF) was prepared in laboratory as process laid down in IS: 848 (1974). All the analytical reagents were procured from SD Fine Chem Limited. Glue line treatment was followed to treat veneers. The compositions of chemicals were added in phenol formaldehyde adhesive on the basis of its solid content just before its application on veneers. The details of chemical compositions and concentrations used for treatment are given below:

1. Boric acid (H_3BO_3) (T_1)- 2, 3 and 4%
2. Silicic acid [$Si(OH)_4$] (T_2)- 2, 3 and 4%
3. Boric acid: Silicic acid (2:1) (T_3)- 2, 3 and 4%
4. Boric acid: Silicic acid (3:1) (T_4)- 2, 3 and 4%

The chemicals as discussed above along with adhesive were applied on both side of core veneer by brush at rate of 110g/m^2. Three veneers were assembled perpendicular to each other and then hot pressed for 8 minutes at 150 ^{0}C and 200 lbs inch^{-2} pressure. After that prepared plywood was subjected to conditioning for 24 hours at room temperature.

Testing

The plywood made of poplar was subjected to white and brown rot to evaluate its resistance through soil block bioassay. The compatibility of the test chemicals were also determined by glue shear strength test at dry, wet and after mycological test. The details are as below:

Soil block bioassay

The samples of size 1.9 x 1.9 x 0.45 cm^3 were prepared from untreated and treated plywood. Samples were marked and their initial weight was noted (W_1). Then samples were subjected to oven at 100-105^{o}C temperature and the weight was noted down till a constant weight achieved (W_2). The fungi selected for the present study were *Oligoporus placentus* Murr and *Trametes versicolor* Linn (IS: 4873, 2008; ASTM, 1980).

Preparation of soil culture bottles

Sieved, air-dried soil of 125 g with pH 5.0-7.0 was filled in screw capped bottles. Distilled water was added to the bottles to maintain moisture of soil in test bottles. Two feeder blocks of size 0.4x1.9x3.5 cm prepared from sap wood of *Bombax ceiba* were placed on the surface of the soil in bottles. Then the bottles with caps (loosened) were sterilized in an autoclave at a pressure of 1 kg/cm^2 for 30 minutes.

Preparation of test culture

Sterilized bottles were thoroughly cooled. The fungus inoculums were taken from freshly grown culture and placed on the edge of the feeder blocks. The inoculated bottles were incubated in B.O.D. (Biochemical oxygen demand) at 25±2^{o}C and 70±4% relative humidity for 21 days or till the feeder blocks were completely covered by the test fungi.

Introduction and incubation of the samples in culture bottles: Two samples were placed on feeder blocks in contact with mycelium in each culture bottle. Then the prepared bottles were incubated for a period of 14 weeks in the incubator at 25±2^{o}C and about 70±4% relative humidity (IS: 4873, 2008). After the incubation period the samples were taken out from the culture bottles, cleaned off to remove mycelium on surface by brush. The samples were dried at room temperature for 3-4 days and after that in the oven till the constant weight (W_3) was obtained.

Calculation of weight loss: Weight loss (%) of samples was calculated from the conditioned weight of the samples before and after testing.

$$\text{Weight loss (\%)} = \frac{W_2 - W_3}{W_2} X100$$

Where W_2 = Conditioned weight of the sample before test
W_3 = Conditioned weight of the sample after test

Compatibility testing of plywood

The compatibility of chemical compositions with PF adhesive was determined by evaluating glue shear strength of plywood. Samples of size 15 cm x 2.5 cm x 0.45 cm^3 were prepared from treated and untreated plywood for standard shear test as per IS: 1734 (Part 4, 1983). Each sample was held at two ends in the jaws of universal testing machine and pulled apart. The grain of sample was kept perpendicular to the direction of application of load. Load was given at rate of 2.5 mm/min during the test. At the time of complete failure of sample, the load (Glue shear strength) was recorded and values were compared with values given in Indian Standard for general purpose plywood *i.e.* IS: 848 (1974). Test was carried out at dry and wet state and after mycological test. For wet state, samples were submerged in boiling water for 8 hours and after removal cooled down at room temperature and tested for shear strength to determine boiling water resistance as per IS: 1734 (1983).

Mycological test was also carried out to evaluate the resistance of glue line to attack by test fungi *i.e.* white rot (*T. versicolor*) and brown rot (*O. placentus*) as per IS: 1734 (1983). A flat rectangular dish of depth 50 mm was half filled with sawdust of semul (*Bombax ceiba*). The sawdust was moistened with water containing 30 g of commercial malt to a litre of water. The saw dust was introduced with the spores of *T. versicolor* and *O. placentus* and loosely compacted. Samples were placed down into it. The dish was covered with a glass sheet and the edges of the dish were sealed. The dish was kept at a temperature of 27 ±2°C for a period of 21 days. After removal, samples were tested for glue shear strength and wood failure percent as IS: 1734 (1983).

Statistical analysis

The data recorded was statistical analyzed using "SPSS" package (16.0) to find out the variation between the treatments and the relationship between the observed parameters. Critical difference (CD) was calculated to determine the variation between means at 5% significance level.

RESULTS & DISCUSSION

Soil block bioassay test

Table 1 exhibits the performance of veneers against *T. versicolor* and *O. placentus* in terms of mean percent weight loss of plywood samples. Results exhibited 34.76 and 28.46% mean weight loss in untreated control sets due to *T. versicolor* and *O. placentus* respectively. Mean weight loss in samples treated with T_1 ranged 17.79-23.58 and 16.39- 21.83 % against *T. versicolor* and *O. placentus* respectively. It showed that T_1 provided sufficient protection to plywood against test fungi as compared to untreated specimens. Weight loss was further reduced with increasing concentration of T_1. It is because of anti-fungal properties of boric acid against wood decaying fungi (Yalincilic *et al.,* 1999). However the protection obtained by glue line treatment with boric acid against the same fungi was lower than veneer treatment (Bhatt *et al.,* 2016).

TABLE 1: Mean weight loss (%) of samples subjected to decay fungi through Soil Block Bioassay

Treatment	Concentration (%)	Mean weight loss (%)	
		T. versicolor	*O. placentus*
Control	-	34.76	28.46
Boric acid (T_1)	2	23.58	21.83
	3	21.19	17.83
	4	17.79	16.39
Silicic acid (T_2)	2	30.23	25.56
	3	27.86	25.27
	4	25.15	20.19
Boric acid: silicic acid (2:1) (T_3)	2	21.66	19.21
	3	18.12	17.98
	4	14.66	13.28
Boric acid: silicic acid (3:1) (T_4)	2	18.98	18.69
	3	16.56	15.27
	4	12.12	11.98

Mean Treatment: Control=31.61%, T_1=19.76%, T_2=25.71%, T_3=17.48%, T_4= 15.60%
Mean concentration: 2%=22.46%, 3%=20.01%, 4%= 16.44%
CD $_{(0.05)}$ Treatment =1.24, Concentration =0.96, Fungi=0.79

Samples treated with T_2 showed 25.15- 30.23% mean weight loss by *T. versicolor* and 20.19-25.56% by *O. placentus*. Although the mean weight loss percent recorded by T_2 was less than untreated, the results are not satisfactory. The results are in conformity with Yamaguchi (2005), who reported that wood treated with silicic acid exhibited no protection against brown rot (*Fomitopsis palustris)*. Mean weight loss in T_3 treated samples was found 14.66-21.66 % against to *T. versicolor* and 13.28-19.21 % against to *O. placentus,* which is 37-57% and 32-54% less than untreated against *T. versicolor* and *O. placentus* respectively. Low weight loss could be noticed in samples treated by $T_{4;}$ exhibiting only 12.12-18.98% and 11.98-18.69% mean weight loss against *T. versicolor* and *O. placentus* respectively. The increased efficacy may because of increased boron content in combinations. Since it was noted that silicic acid used in combination with boric acid exhibited less weight loss as compared to silicic acid alone. Similar studies have been reported in recent past, which had recommended the use of boric acid with silicic acid (Saka *et al.,* 1999). Yamaguchi (2002) also reported decay resistivity of silicic acid monomer aqueous solution combined with boric acid against *Coriolus versicolor* (white rot) and *Tyromyces palustris* (brown rot). In another study Furuno *et al.,* (1992) reported the efficacy of polysilicic acid with boric acid and borax against fungal decay. The combinations of boron with tetra ethoxy silane and methyl tri ethoxy silane increased the decay resistance against *Fomitopsis palustris* and *Trametes versicolor* when compared to untreated (Kartal *et al.,* 2009).Therefore, it is inferred that the increased quantity of boron in combination imparted more protection to samples against test fungi. From the CD value, it was inferred that statistically there is a significant difference among the activity of treatments and concentration in terms of weight loss (%) of test fungi ($p \leq 0.05$).

T. versicolor has caused significantly high decay in samples as compared to *O. placentus* ($p \leq 0.05$). The possible reason may be the similar molecular structure of phenol formaldehyde adhesive and lignin reported by Gusse *et al.,* (2006). It is stated that white rot generally attacks lignin as well as cellulose and the molecular structure of lignin is same as phenol formaldehyde adhesive, due to which white rot may deconstruct the phenolic adhesive same as lignin.

Compatibility test

Glue shear strength (GSS) at dry and wet state

Mean GSS recorded in treated as well as untreated (control) plywood samples at dry and wet state are exhibited in Table 2. As per IS: 848 (1974), the mean shear strength for general purpose plywood should be minimum 135 and 100 Kg in dry and wet state respectively. Untreated samples showed mean GSS of 140.70 and 113.70 Kg at dry and wet state respectively. The adverse effect of boric acid (T_1) on GSS was observed in dry and wet state, since mean GSS was recorded less than 135 and 100 Kg in dry and wet state respectively (Table 2). Shukla (1991) also reported ill effect of boric acid on the glue shear strength of plywood of *Populus deltoides.* In previous study done by Kartal *et al.* (2008), the preservatives contain boron such as didecyl dimethyl ammonium tetrafluoroborate (DBF) affected the bonding performance of phenol formaldehyde adhesive (Basically, boron ions react with the functional methylol groups on the resin molecules before the curing of adhesive and affect the bond strength (Chai *et al.,* 2016).

Samples treated with silicic acid (T_2) showed 136.00-143.70 Kg and 107.70-115.50 Kg means GSS at dry and wet state respectively, which shows the compatibility of silicic acid with PF adhesive in dry as well as at wet state. The results are in conformity with the findings reported by Bhatt *et al.,* (2015). Mai and Militz (2004) reported silicon as adhesion promoters, surface modifiers and cross linking agent. Burton and Deale (1996) elaborated silicic acid as low volatile weak acid, which helps in curing of adhesive. Samples treated with the combination of boric and silicic acid at 2:1 ratio (T_3) showed satisfactory results at all state i.e. dry and wet, whereas, the combination at 3:1 ratio (T_4) could not exhibited satisfactory results at both state (Table 2). It was found that shear value decreased with increasing concentration of the T_4. It may because of increasing concentration of boron in the combination, which showed adverse affect on shear values. Analysis revealed significant effect of treatment on shear strength in both dry and wet state, whereas 2 and 3 % concentration showed similar results ($p \leq 0.05$).

TABLE 2: Mean glue shear strength (Kg) of plywood samples at dry and wet state

Treatment	Concentration (%)	Mean glue shear strength (Kg)	
		Dry	Wet
Control	-	140.70	113.70
Boric acid (T_1)	2	130.80	95.40
	3	128.40	90.30
	4	114.56	81.00
Silicic acid (T_2)	2	136.00	107.70
	3	138.60	108.06
	4	143.70	115.50
Boric acid: silicic acid (2:1) (T_3)	2	138.90	104.40
	3	136.80	100.13
	4	135.90	99.35
Boric acid: silicic acid (3:1) (T_4)	2	133.76	97.96
	3	131.90	95.16
	4	122.70	90.90

Dry state
Mean Treatment: Control=140.7Kg, T_1=124.6 Kg, T_2=139.4 Kg, T_3=137.2 Kg, T_4= 129.5 Kg
Mean concentration: 2%= 134.9 Kg, 3%= 133.9 Kg, 4%= 129.23Kg
CD $_{(0.05)}$ Treatment =3.78, Concentration = 2.93
Wet state
Mean Treatment: Control=113.7Kg, T_1=88.9 Kg, T_2=110.6 Kg, T_3=101.5 Kg, T_4= 94.6 Kg
Mean concentration: 2%= 101.4 Kg, 3%= 98.7 Kg, 4%= 96.8 Kg
CD $_{(0.05)}$ Treatment =3.35, Concentration =2.60

TABLE 3: Mean glue shear strength (Kg) of plywood samples after mycological test against fungi

Treatment	Concentration (%)	*T. versicolor*		*O. placentus*	
		Mean GSS (Kg)	Wood failure (%)	Mean GSS (Kg)	Wood failure (%)
Control	-	100.80	70	114.30	60
Boric acid (T_1)	2	104.70	35	107.10	30
	3	100.20	30	102.00	25
	4	100.20	20	100.08	20
Silicic acid (T_2)	2	102.00	70	103.50	60
	3	105.30	50	107.10	45
	4	106.43	50	109.50	40
Boric acid: silicic acid (2:1) (T_3)	2	105.90	30	113.70	30
	3	104.70	30	108.30	25
	4	103.50	25	104.70	20
Boric acid: silicic acid (3:1) (T_4)	2	117.00	30	122.23	25
	3	112.80	25	118.53	20
	4	108.20	20	117.90	10

Mean Treatment: Control=107.6Kg, T_1=102.6 Kg, T_2=105.80 Kg, T_3=106.9 Kg, T_4= 116.2 Kg
Mean concentration: 2%= 109.5 Kg, 3%= 107.5 Kg, 4%= 106.6 Kg
Mean fungi*:* *T. versicolor* = 105.6 Kg, *O. placentus*=110.1 Kg
CD $_{(0.05)}$ Treatment =3.42, Concentration=2.64, Fungi=2.16

Glue shear strength after mycological test

Untreated samples revealed mean GSS of 100.80 and 114.30 Kg against *T. versicolor* and *O. placentus* respectively and 60 to 70% wood failure. High values of wood failure in untreated samples indicated the rupturing of wood during GSS testing, which may be due to poor resistance of untreated samples against test fungi in mycological test. However, untreated samples fulfilled the criteria of general grade plywood as per IS: 848 (1976). Samples treated with T_1 showed 100.20-104.70 Kg and 100.08-107.10 Kg mean GSS against *T. versicolor* and *O. placentus* respectively and wood failure percentage ranged from 20-35%. The low values of wood failure indicate the high protection of veneers imparted by T_1 against test fungi, as boric acid is well established wood preservative. Mycological test with T_2 showed low mean GSS values ranged from 102.00 to 106.43 Kg and 103.50 to 109.50 Kg against *T. versicolor* and *O. placentus* respectively, whereas, high wood failure percent ranged from 40-70% was recorded with T_2. The results of soil block bioassay test also showed low efficacy of T_2 against both the test fungi. Therefore, it may be assumed that silicic acid could not protect veneers effectively from the test fungi and resulted low GSS with high wood failure.

T_3 showed mean GSS of 103.50-105.90 Kg and 104.70-113.70 Kg against *T. versicolor* and *O. placentus* respectively with 20-30% wood failure. Treatment with T_4 also exhibited high GSS and low wood failure percent in mycological test (Table 3), which indicates effective bonding of veneers. Overall the best results are observed by T_4 followed by T_3, T_2 and T_1. Statistically T_2 and T_3 exhibited similar shear strength as untreated after mycological test ($p \leq 0.05$). It is difficult to ascertain the reason for this observation and results. However, the probable reason may be use of high amount of boron in combination, since the wood failure reduced with increasing amount of boron in combination. It is interesting to note that the effect of wet state on shear strength is more pronounced than mycological attack in plywood (Table 2 and 3).

CONCLUSION

The results show that boric acid is effective as preservative against both decaying fungi, but at the same time adverse effect on shear strength was observed which rejected its suitability for poplar plywood. The results showed that samples treated with silicic acid met the minimum requirement of general purpose plywood as per IS: 848 (1974), but results of soil block test was not satisfactory. It is concluded that silicic acid had no antifungal activity; hence collectively silicic acid cannot be suggested for plywood treatment. Although the combinations of boric and silicic acid imparted the resistance to test fungi by reducing the weight loss as compared to untreated samples. However, only T_3 produced satisfactory glue bond strength and passed the minimum shear measures for general grade plywood as mentioned in IS: 848 (1974). It indicates that boric acid has certain interaction with silicic acid, which contribute towards both compatibility and decay resistance in plywood in 2:1 ratio. The present study exhibited the potentials of the combination of silicic acid and boric acid as such, which was not reported earlier specifically for plywood to provide sufficient durability. It can provide sufficient protection to plywood against test fungi without affecting its shear strength. Another advantage, of silicic acid is to limit the boron leach- ability and making it available for wood protection against decaying fungi.

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