

# Composite Building Panels from Local Resources

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## Abstract

Composite panels were made from locally available pine needles and isocyanate prepolymer adhesive. Prior to use, wettability of the pine needles treated under various conditions (alkali, steam and alkali-steam combination) was assessed through contact angle measurement. It was found that alkali-steam treated pine needle fibres were more wettable than that of the other treatments. The physico-mechanical properties and performance characteristics of these composite panels were studied with respect to resin content, needle fibre length and pine needle-wood particle ratio. The composites containing 5% resin content and 3 mm furnish size and also pine needle/wood particles in 50:50 ratio exhibited acceptable properties meeting the requirements of the commercial standard specification. It was noted that the pine needle/wood particle composites exhibited more equilibrium moisture content than the pine needle composites showing their less dimensional stability. Flammability results indicate that the total heat release, effective heat of combustion and total smoke release in the flaming mode reduced significantly when pine needles were treated with the urea phosphate solution. Reaction to fire characteristics such as fire propagation index, surface spread of flame and smoke density were also assessed according to BS: 476 to know contribution of samples towards fire growth. A pilot plant trial on the manufacturing of pine needle composite panels was made for their commercial exploitation.

**Keywords: Pine needles, Composites, Wettability, Dimensional stability, Flammability**

## 1. Introduction

Interest in using non-woody ligno-cellulosic materials such as stalks and leaves of plants and agro-forest residues has received considerable attention in the industry for producing panels, door inserts, core materials for sandwich construction, furniture etc (Rowell 1995; Yalinkilic et al. 1998; Guler et al. 2006 and Nemli et al. 2008). These non-woody materials have desirable physical properties and also exhibiting ligno-cellulosic similarity with the wood. However, one of the most critical factors is their outer enriched waxy layer that restricts bondability with resin adhesives. To overcome these shortcomings, various attempts have been made either to increase non-wax bonding surface area by mechanical / chemical treatment (Schmidt et al. 2002 and Zheng et al. 2007) or by use of newer adhesives (Papadopoulos et al. 2002 and Bisanda et al. 2003). With commonly used formaldehyde based resins, the non-wood based panels exhibited acceptable physical and mechanical properties to produce specification grade products. Concerns are, however,

raised on human health due to formaldehyde emission from these panels in view of their 10-15% annual global market demand. Polyisocyanates are being considered as the appropriate binder over the conventionally used formaldehyde based resins for waxy ligno-cellulosics in terms of tolerance to high fibre moisture content, low temperature processing, low resin doses, high spread rate and low press pre-cure time (Walsh 1994). But the isocyanate bonded materials stuck to caul plates and other metal surfaces during composite panels manufacturing. The use of polyisocyanate adhesives as bonding agent is still a point of debate on a commercial scale due to optimization of materials, processes and cost involved at the production stage. This necessitates research focus on searching of newer adhesive binders in making dimensionally stable ligno-cellulosic panel products for building applications.

In the present study, the pine needles were used as raw material alternative to wood flakes/particles for making panel products because of their renewableness and availability in the local area in large quantity (2.7 million tones / annum). Isocyanate prepolymer was selected over conventionally used formaldehyde based resins and polyisocyanates. In earlier studies (Coleman and Biblis 1977; Piao et al. 2004 and Nemli et al. 2008), attempts on utilization of pine needles were made for making particle boards/fibre boards. Because of waxy surface of the pine needles, the resultant boards exhibited weak surface strength, poor dimensional stability and low internal bond strength. In order to overcome some of these shortcomings, several methods such as pretreatment of pine needles (alkali, acylation etc.), blending of pine needles with other fibres (bamboo and wood species) and composite resin adhesives (urea formaldehyde and polyisocyanate) were adopted to produce dimensionally stable building boards/panels (Piao et al. 2004 and Nemli et al. 2008). Efforts on pilot plant level trials for producing pine needle composite boards/panels are yet to be made to understand their commercial viability.

In this paper, wettability of the pine needles treated under different conditions was studied by the contact angle method. The performance of composites made from pine needles / isocyanate prepolymer was reported in terms of their physico-mechanical properties, dimensional stability and flammability characteristics. Based on optimized materials parameters, a pilot plant study has been conducted on a commercial unit to manufacture pine needle composite panels. The prepared panels were also assessed as per existing standard specifications.

## **2. Experimental**

### **2.1. Materials**

The pine needles of 300-380 mm length were collected from the Indian forests (density, 0.22 g/cm<sup>3</sup>; moisture content, ~20%; water absorption, ~45%). The needles were comprised of cellulose (40-43%), hemicellulose (20-24%), lignin (36-40%) and ash content (2-4%). Aromatic polyisocyanate prepolymer was obtained from M/s Bayer Material Science Pvt. Ltd. India (Desmodur E 23 - NCO content, 15.4 ± 0.4%; viscosity, 1800±250 mPa.s; density, 1.13 g/cm<sup>3</sup>). Commercial grade sodium hydroxide and urea phosphate were obtained from local market.

## **2.2. Fabrication of composite panels**

Pine needles were cut to a desired length (~ 30 mm). Thereafter, they were treated with 2 wt% aqueous sodium hydroxide solution (needles). Subsequently, samples were dried and hammer milled to a furnish size upto 3 mm. Composite panels were prepared using untreated/treated pine needle furnishes and isocyanate prepolymer adhesive (3 - 20 wt%) on a hydraulic press at 140°C and 10 MPa pressure for 5 min retention. Before pressing, the resin was sprayed on to needle furnishes and mixed in a blender. Subsequently, the mix was laid on a silicone paper lined mould in the form of a mat. The mould was allowed to cool at room temperature. Fire resistant composite boards were also prepared with urea phosphate treated needle furnishes.

## **2.3. Methods**

### **2.3.1. Physico-mechanical Tests**

The physico-mechanical tests such as density, water absorption, thickness swelling, internal bond strength, screw withdrawal strength and flexural strength of the samples were measured as per ASTM D 1037-2006. The flexural properties of samples were tested at a cross-head speed of 5 mm / min and span-to-depth ratio of 16:1. The internal bond strength of samples was determined by testing tensile strength perpendicular to the surface on a Hounsfield material testing machine (H 25 KS) at a cross head speed of 0.08 mm / mm of thickness per min. The screw holding test was carried out at a loading rate of 1.5 mm / min. The wood screws No 8 and 50 mm length were threaded into the specimen at right angle to the face upto a half of their length in a pre-bore of 2.5 mm. All results were the average value of five measurements.

The contact angle of untreated and treated pine needle surfaces was measured using sessile drop technique with the help of a Dynamic contact angle analyzer (VCA Optima XE, AST Products Inc). Surface energy software (SE-2500) was used for calculation of critical surface energy of untreated and treated pine needles using Zisman plots.

The samples were exposed for 60 days in different relative humidity (35%, 75%, 98%RH and 98% RH at 50 °C). The relative humidity was maintained in a desiccator according to the method described in ASTM C 427-1964. Thickness swelling and linear expansion in the exposed samples was recorded at a regular interval of time. The samples were also aged under various alternate wetting/drying to measure their thickness swelling.

### **2.3.2. Flammability tests**

Cone calorimeter (FTT Ltd.) has been used to measure flammability characteristics of the pine needle composite panels according to ISO 5660-1: 2002. The test run was conducted for 20 min at the heat flux of 50 Kw/m<sup>2</sup> and normal duct flow rate of 24 l/s. The fire propagation test on the specimen of size 225 x 225 x 12 mm was conducted as per BS EN 476 -1981(part 6). The test run was continued for twenty minutes duration to record propagation indices. The surface spread of flame test was carried out on the samples of size

270 x 900 x 15 mm according to BS EN 476-1981 (part 7) to know fire class of the samples. The smoke density of samples was measured according to ASTM D 2843-2004 under flaming and non-flaming modes. Rate of burning of pine needle composite boards/panels was determined as per ASTM D 635-2006. The time taken from 30% to 70% weight loss was recorded.

### 3. Results and discussion

#### 3.1. Wettability studies

The pine needles were treated with alkali, steam and combined use of steam and alkali to obtain their adequate bonding with resin adhesives. Wettability characteristics of these untreated and treated pine needles are given in Table 1. The difference in contact angles of the untreated and treated needles is attributed to the removal of extraneous matters from the surfaces due to action of alkali and temperature during treatment.

Low contact angle for the treated needle furnish clearly indicates its superior wettability over the untreated ones. While comparing the efficacy of treatments, it was found that the alkali and steam treated pine needles showed comparable contact angle and work of adhesion values whereas their combination had low contact angle and high work of adhesion over the other treatments. Improved adhesion of the treated needles was confirmed by measuring critical surface tension through Zisman's plot. It was found that alkali and combined alkali-steam treated needles exhibited 23% less critical surface tension than the untreated ones showing more physical roughness. On the contrary, steam treated needles showed comparable critical surface tension to the control. Because of improved wettability and simple treatment procedure, alkali treated needles were used for subsequent experiments.

#### 3.2. Optimization of parameters

The optimization of various parameters such as treating conditions of pine needles, furnish length, adhesive content and pine needle-wood particle ratio in the composites was made

**Table 1: Surface energetic characteristics of untreated and treated pine needles**  
(Standard deviation is given in parenthesis; Surface tension of water :  $72.8 \text{ mN m}^{-1}$ ; surface tension of formamide :  $58.20 \text{ mN m}^{-1}$ )

Property Treatment	Contact angle ( $^{\circ}$ ) Probe liquid		Critical surface energy ( $\text{mJm}^{-2}$ )	Surface-free energy ( $\text{mJm}^{-2}$ )	Polar component ( $\text{mJ m}^{-2}$ )	Dispersive component ( $\text{mJ m}^{-2}$ )
	Water	Formamide				
Control	84.50 (3.38)	64.40 (2.58)	33.50 (1.34)	30.90 (1.24)	5.00 (0.20)	25.90 (1.04)
Alkali	65.60 (2.62)	53.50 (2.14)	25.60 (1.02)	38.10 (1.52)	18.50 (0.74)	19.60 (0.78)
Steam	64.40 (2.58)	49.80 (1.99)	33.90 (1.36)	40.00 (1.60)	17.60 (0.70)	22.40 (0.89)
Alkali steam combination	56.90 (2.28)	46.60 (1.86)	25.80 (1.03)	44.20 (1.77)	25.30 (1.01)	18.90 (0.76)

(Gupta et al.2010 a & b and Monika et al. 2012). The composites made from alkali treated pine needles exhibited superior properties than those of other treatments. The physico-mechanical properties of composites increased with increasing isocyanate prepolymer adhesive content (up to 20 %). It was found that the composite panels at 5% adhesive content satisfied the commercial requirement of the ligno-cellulosic panel products. The screw withdrawal load of composites containing high adhesive content (20%) had a comparable value to the natural wood. Attempts were also made to prepare the modified isocyanate prepolymer adhesive with functionalized lignin keeping this in view to reduce its cost and superior adhesiveness. When this adhesive was used for bonding of pine needles, the resulting panels possessed superior physico-mechanical properties than that of the parent system (Gupta et al. 2010). Improvement in the properties of composite panels was further made by blending pine needles with wood particles of secondary species (eucalyptus). The mechanical properties of these composites increased with increasing wood particle contents. Both physical and mechanical properties of these composites were optimized at pine needle and wood particle in 50:50 ratio (Monika et al. 2013). Table 2 shows properties of the composite panels with different sizes of needle furnish. The composites made with large furnish size (3mm) exhibited higher density than those of others mainly due to high compaction ratio. Because of compaction ratio, the water absorption and thickness swelling of composites was reduced by 17.54% and 20.24% respectively. A moderate increase in the modulus of rupture, modulus of elasticity and screw withdrawal strength of the composite panels with larger furnishes were observed compared to the composites made with the smaller needle furnishes.

The vertical density profile of composite panels as determined by the gravimetric method is shown in Figure 1. The samples exhibited high surface density and a low core density. These variations occurred probably due to the unequal distribution of heat and moisture transfer through the mat during pressing (Kelly 1977). It was noticed that the adhesive in the

**Table 2: Properties of composite panels with different size of pine needle furnishes**

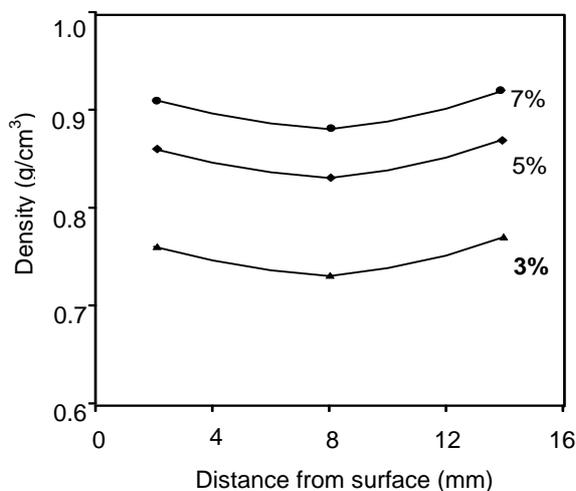
Property	Pine needle composites (average size 2 mm)	Pine needle composites (average size 3 mm)
Density (gm/cc)	0.90 (0.05)	1.15 (0.57)
Water absorption (%)		
2 hrs soaking	19.11 (0.95)	17.50 (0.87)
24 hrs soaking	45.44 (2.27)	37.47 (1.87)
Linear expansion 2 hrs soaking (%)		
Length	0.19 (0.01)	0.17 (0.01)
Width	0.27 (0.02)	0.39 (0.02)
Thickness swelling 2 hrs soaking (%)	12.60 (0.63)	10.05 (0.50)
Modulus of rupture (MPa)	20.20 (1.01)	21.19 (1.06)
Modulus of elasticity (MPa)	1361.52 (68.08)	2644.14 (132.21)
Internal bond strength (MPa)	1.12 (0.06)	1.20 (0.06)
Screw withdrawal strength Face(N)	1270 (63.50)	1365.90 (68.29)

core layer was also in a pre-cured state due to insufficient temperature/ humidity. Because of vertical density gradient, the composite panels exhibited unbalanced dimensional changes with respect to the humidity. When pine needles were blended with wood particles, the difference in the density of the top and core layers was minimized. This could be expected to the adhesive curing of core layers due to availability of more moisture from wood particles than the needle furnishes.

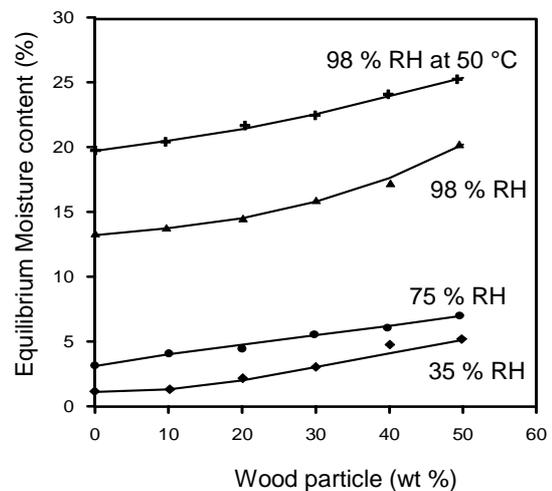
### 3.2.1. Hygro/Hydro thermal aging

The composite samples were exposed under various humidity and alternate wetting/drying cycles to know their dimensional stability under wet environment. As the humidity level increased, the equilibrium moisture content of samples also increased (Fig 2). The samples exposed under hygrothermal condition (98 % RH/50 °C) had more equilibrium moisture content (25 %) than the samples exposed under high humidity alone (98 % RH) attributable to the combined effect of moisture and temperature. It was noted that the equilibrium moisture content of pine needle composites increased with increasing wood particle content probably due to the hydrophilic surface of wood particles. At low humidity (upto 75 %),the thickness swelling in the samples ranged between 3-6 % at equilibrium moisture content whereas at high humidity, the maximum thickness swelling was ~20 %. The samples experienced more thickness swelling (25 %) when these were subjected to hygrothermal condition (98 % RH/50 °C). The linear expansion of the samples increased with increasing humidity (Fig 3). At low resin content (3 wt%), the linear expansion in the samples was ~ 1% under high humidity whereas at high resin content (20 %), the linear expansion remained ~ 0.2 % only because of adequate coating of resin on the surface of the particles and also good particle-particle bonding.

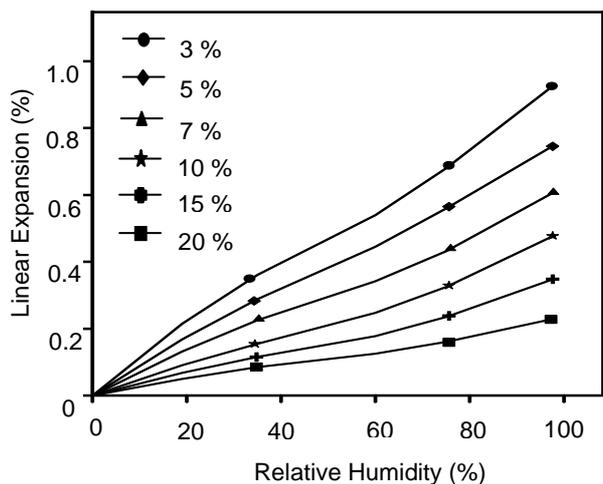
Figure 4 shows thickness swelling of composite samples under various hydrothermal conditions. The samples were immersed in cold water for 24 hrs, 2 hrs immersed in water at 70 °C and 2 hrs immersed in water at 100 °C and these were followed by drying at 50 °C to a constant moisture content (Sumardi et al. 2007). The samples containing 3 % resin content affected more than those of others mainly because of bond disruption between the



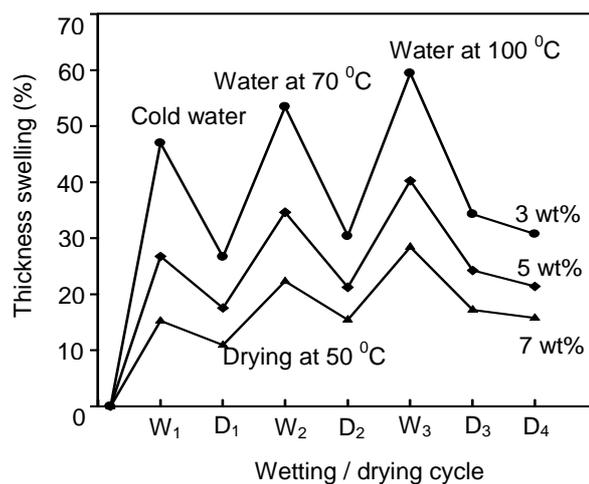
**Figure 1: Vertical density profile of pine needle composite panels with different resin content**



**Figure 2: Effect of wood particle content on equilibrium moisture content of pine needle composites under various humidity**



**Figure 3: Effect of humidity on the linear expansion of pine needle composites at different resin content**



**Figure 4: Thickness swelling of composite samples with different resin contents exposed under various exposure cycles**

fibre and resin as a result of expansion and contraction of fibres. The thickness swelling occurred in the range of 45-60 %. When resin content in the composites increased from 3 to 7 %, the thickness swelling was reduced by two-three times. It was noted that the samples were more dimensionally unstable when these were aged at 100 °C for 2hrs.

### 3.2.2. Fire behavior

The cone calorimetric flammability data for various pine needle composite panels are given in Table 3. The total heat release of these panels ranged between 37 mJ/m<sup>2</sup> to 41 mJ/m<sup>2</sup>. When urea phosphate treated needles were used in the panels, a decrease of ~ 80 % in the total heat release was observed. The rate of heat release reduced to 2.82 Kw/m<sup>2</sup> only compared to 10 Kw/m<sup>2</sup> of the control. The specific extinction area at the end of fire was also negligible probably due to non-availability of air/fuel. The sample containing treated pine needles seems to be less combustible as the value of effective heat of combustion was very small (0.29 mJ/Kg) compared to 8 - 10 mJ/Kg for the untreated ones. The release of low heat from the samples would be expected to reduce their ignitability characteristics and consequently, the growth of the fire. It was also noted that the samples containing treated needles produced more smoke than those of others under non-flaming mode. In the smoke, carbon dioxide yield was less and carbon monoxide yield was more compared to the gases released from the samples made with untreated needles due to the existence of fire retardant chemical.

Based on the above screening results, the composite panels made with the treated pine needles were assessed for their reaction to fire characteristics according to BS: 476. As seen in the time-temperature curve (Figure 5), a steady state was reached after 15 min of fire growth during fire propagation test. A substantial temperature difference between the reference and sample was noticed after 5 min of onset of ignition. The value of fire propagation indices of samples calculated from the curve were 5.67 (initial) and 17.52 (total) respectively. These values were comparatively less than the other materials such as particle boards (36.52), fibre board (56.09), plywood (38.77) and wood (41.15). A material having a

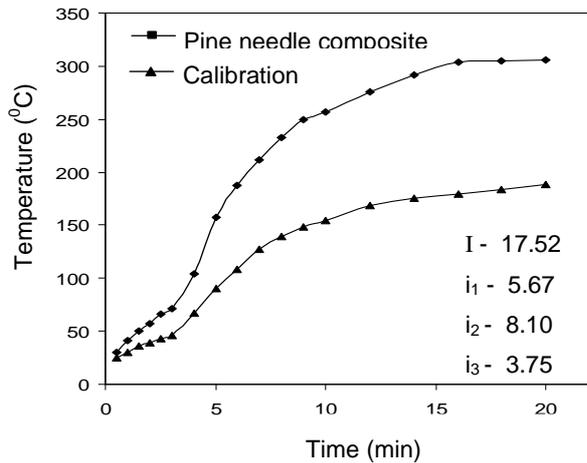
**Table 3: Cone Calorimetry results of the Untreated & Fire retardant treated Pine needle boards**

Property	Pine needle board	Pine needle : wood particle board	Fire retardant treated pine needle board
Heat release rate (KW/m <sup>2</sup> )	11.12 (0.56)	10.49 (0.52)	2.82 (0.14)
Total Heat release (mJ/m <sup>2</sup> )	40.60 (2.03)	37.80 (1.89)	5.90 (0.29)
Average specific mass loss rate(g/s/m <sup>2</sup> )	10.28 (0.51)	11.90 (0.59)	9.39 (0.47)
Effect heat of combustion (mJ/Kg)	10.52 (0.53)	8.41 (0.42)	0.29 (0.01)
Specific extinction area (m <sup>2</sup> /Kg)	78.33 (3.92)	52.51 (2.62)	-135.38 (-6.77)
CO <sub>2</sub> Yield (Kg/Kg)	1.15 (0.06)	1.09 (0.05)	0.39 (0.02)
CO yield (Kg/Kg)	0.0071 (0.0003)	0.0097 (0.0005)	0.1011 (0.005)
Total Smoke release (m <sup>2</sup> /m <sup>2</sup> )			
Non-flaming phase	2.10 (0.11)	2.80 (0.14)	8.30 (0.42)
Flaming phase	408.10 (20.41)	370.90 (18.55)	94.00 (4.70)

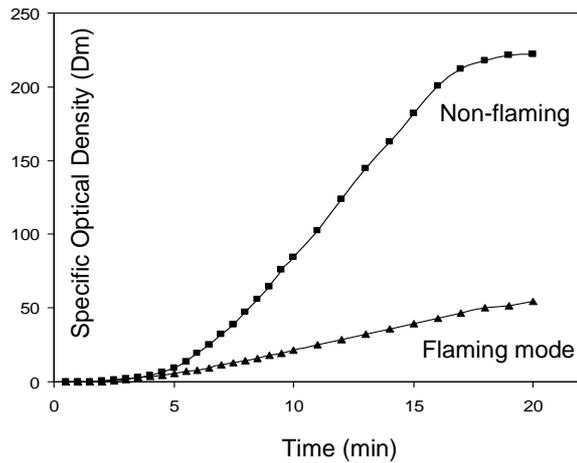
higher index can be considered to contribute more to fire growth than one of lower index. During surface spread of flame test (BS: 476 Part 7), the samples were charred at contact point of igniting flame. The cracks were appeared on the surface. The time of spread of flame for specified length (165 ± 50 mm) was insignificant. Based on the extent and rate of flame spread observation, the samples were classified into Class I i.e. surfaces of very low spread of flame. In supplement to cone calorimetry, the specific optical density of samples was also tested in a smoke density chamber. It was observed that after 12 min of radiant heat exposure, a sudden rise of specific optical density (221.2 Dm) in the non-flaming mode was observed (Fig 6). In the flaming mode (radiant heat and flame), the specific optical density of the samples was found to be 54.19 Dm only. In both modes, these values are comparatively less than the wood (Flaming: 228 Dm and non-flaming: 328.70 Dm). The observations on smoke release were similar to the results obtained from the cone calorimetry measurement. This property is related to the percentage reduction of light flux due to smoke across a light path and causes a reduction in visibility. Under burning test, there was a steep rise of weight loss as a function of time. The sample lost 80 % of its weight after 40 min of exposure. The rate of burning of samples was 2.1 %/min.

### 3.3. Pilot plant study

Industrial trials on the production of the pine needle composite panels were made on a commercial unit. Various processing parameters such as resin efficiency, mat moisture content, furnish size and pressing conditions were examined to achieve satisfactory products at minimum cost. The optimum resin flow was obtained from a viscosity-temperature curve (Fig 7). Viewing the minimal variation in the viscosity after 110 °C, the press temperature and pressure of the panels were adjusted to obtain adequate resin spread onto needle furnishes. It is likely to mention that all particles are not in an intimate contact with neighboring particles over their entire surface after mat consolidation in the press. Because of this, high pressure and resin droplet through fine atomization in the blender were adopted. The properties of pine needles depend on their acidity, extractive

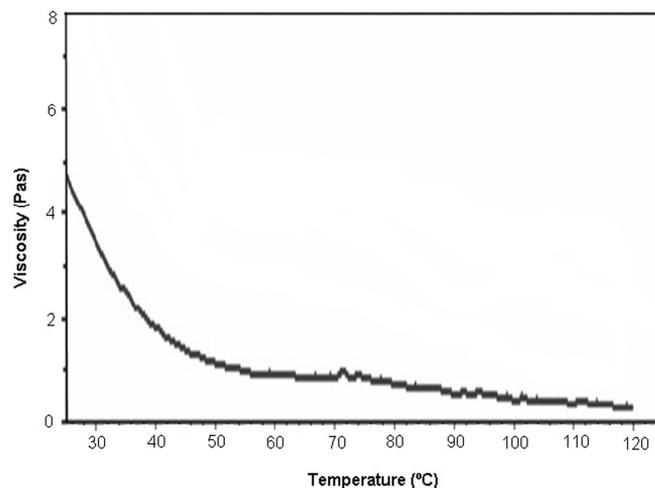


**Figure 5: Time-temperature curves under fire propagation test**



**Figure 6: Smoke density of samples**

contents and machinability. During milling, the pine needles were produced in a non-uniform size. Two average particle sizes of needle furnish (2 mm and 3 mm) were selected in the preparation of composite panels at the same resin level. Because of high surface area coverage of fine particles, it was observed that the total adhesive requirement to bond individual particle was more resulting in inferior panels properties as compared to large size furnish (3 mm). Extractives leached out from the pine needles is expected to serve as a dimensional stabilizing agent when these were reacted with isocyanate minimizing water absorption and thickness swelling of the composite panels. Due to low density of needle furnish, the high compaction ratio (density of particle board/ density of furnish) was used to obtain satisfactory performance. The mat moisture content below 20 % is essential for the reactivity with isocyanate. Using this resin, the press cycle shortened contrary to formaldehyde base resins. The central region of the panel is always at the lowest temperature. Therefore, it was adjusted in such a way to ensure that core reaches a sufficient high temperature to allow the resin to cure. The faster press closing with 5 min retention was selected to obtain desired vertical density profile.



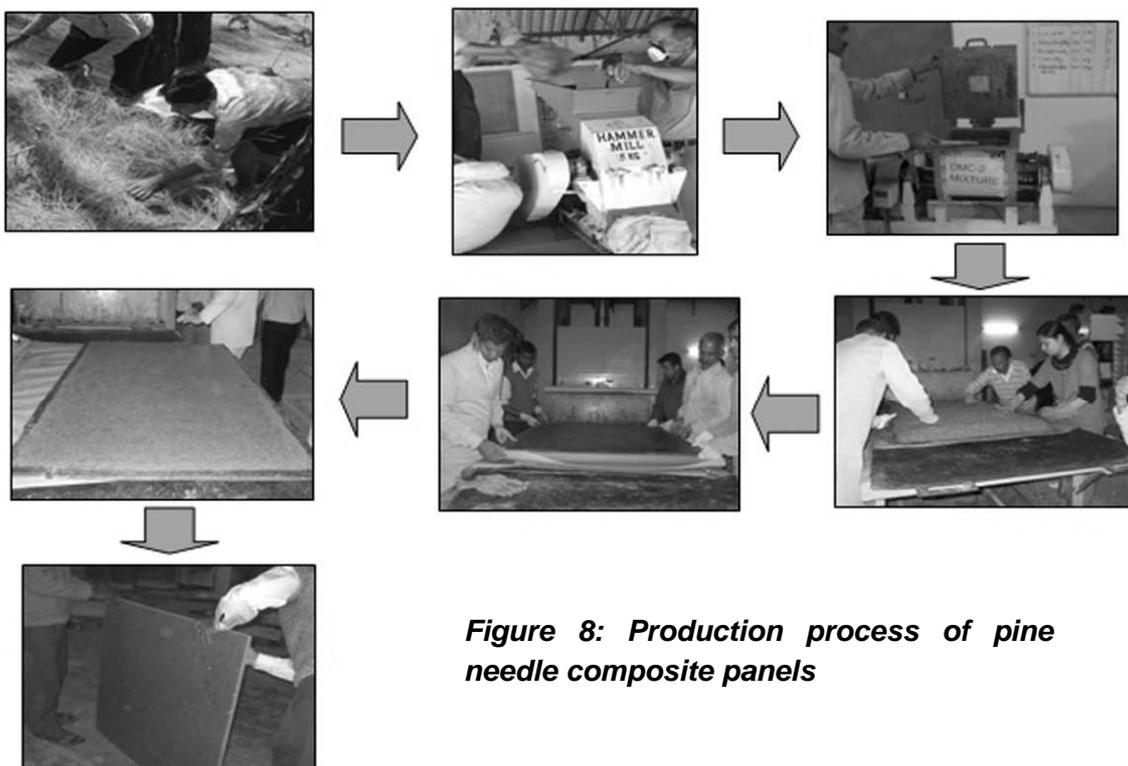
**Figure 7: Viscosity versus temperature curve of isocyanate prepolymer at a shear rate of 2 /s at 25 °C**

For industrial trials, about 200 Kg of pine needles were collected from the Indian forests. These needles were chipped and hammer milled preferably to a size of 3 mm. Subsequently, the needles were treated for 2 hrs in the sodium hydroxide solution at room temperature in a plastic lined tank. After treatment, the alkali from the pine needles was removed by washing with the running water and then, dried in a rotary dryer. The needle furnishes (~ 40 Kg) were mixed with isocyanate prepolymer (2-3 Kg) in a blender. The needle furnish mats were prepared on a mould plate. These mats were stacked over each other and pressed in a single daylight press (300 tons capacity) at 130-140 °C, 15 - 20 MPa pressure and retention time ~ 5 min. The demoulded panels were trimmed. Various sizes of panels such as 1.2 m x 1 m and 2 m x 1 m were prepared and tested for various properties. The photoview of some manufacturing steps are shown in Figure 8. The cost of panels is ~6 \$US / m<sup>2</sup> and comparable with the commercial panels.

The salient features of pine needle composite panels are: dimensionally stable, sufficient internal bond strength, adequate retention of properties under wet / humid conditions, easily cut and sawn, good screw holding strength, good sound and thermal insulation and easily laminated. The composite panels belong to medium and high density boards categories (density 0.7-1.2 g/cm<sup>3</sup>) and meets the requirements of Standard Specification (Bureau of Indian Standards 2005).

#### 4. Conclusions

The results indicate that pine needles in a processed state can be effectively utilized in the manufacturing of composite boards/panels in view of increasing shortage of wood resources. The isocyanate prepolymer is suggested over the conventional adhesives (urea formaldehyde and polyisocyanates) for bonding the pine needles due to its good



**Figure 8: Production process of pine needle composite panels**

penetrability into waxy surfaces and inter-particle bonding. Because of their weak strength, the pine needles can also be blended with other ligno-cellulosic fibres in producing composite panels of acceptable properties. Fire retardancy in the composite panels can be provided by treating needle furnishes with urea phosphate. The improved vertical density profile of panels can be obtained by adjusting an optimum processing condition. Resin flow (spreading) obtained under viscosity-temperature curve was used to finalize pressing temperature and pressing time. For making a commercial viable, a pilot plant study on the manufacture of composite panels was made. Various panels manufactured during the process satisfied the commercial requirements. The developed panels can be used as boards, partitions, door inserts, door skins and furniture items. Under humidity and hydrothermal conditions, the composites containing 5% resin content are dimensionally stable and exhibited less water absorption, thickness swelling and internal bond strength than the acceptable criteria of commercial specification. It is expected that service life of panels may be 10 -15 years even under wet environment.

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