Sinha ASK

Kumar Mohit

[1]Department of Chemical Engineering,
Sant Longowal Institute of Engineering & Technology,
Longowal-148106, Punjab, India

Study Of Chemical, Mechanical And Thermal Properties Of Wheat Straw Fibers And Phenol Formaldehyde Composite

Abstract

Polymeric materials have replaced majority of the conventional metals and materials in various fields in last few years. In this research, wheat straw fibres obtained by soda pulping have been used as reinforced material and phenol formaldehyde resin (resole type) as matrix for manufacturing the composite. The weight ratio of fibres to resin has been maintained at 40:60. Phenol formaldehyde resin is a good adhesive in nature and binds fibres tightly to each others. The effect of different process parameters such as temperature (120 160^{0}C), pressure (5.16 27.54 MPa), and time (15 60 minutes) on composite properties have been studied. These properties are tensile strength, impact strength, water resistance, swelling test and differential scanning calorimetric test. Phenol formaldehyde composites showed the optimum properties at 140^{0}C temperature, 27.54 MPa pressure, and 45 minutes curing time. Phenol formaldehyde composite has good water absorption resistance in acidic and neutral medium, but shows poor resistance in alkaline medium. This provides environmental friendly product which was shown by DSC test results that the composite could be degraded at 183 ^{0}C.

Key words: *Phenol formaldehyde, Wheat straw fibres, Composite, Tensile strength, Impact strength.*

Introduction

The objective of this research work is to find some natural fibrous product which has high mechanical strength and water resistance but easy to degrade resulting in environment friendly material. The polymeric products like polyethylene (LDPE, HDPE), polypropylene, PETs and other composites have replaced the older easily recyclable materials like paper and paper boards, metal containers used for packaging and mechanical support in different industrial applications. But the main problem is environmental pollution caused due to difficulty in their degradation and piling of huge quantities of waste plastic materials. So there is lot of demand for research and development of biodegradable high strength materials which have easy degradation and use natural fibers obtained from recyclable resources like wheat and rice straw as important ingredient. The most important advantages of the natural fiber based polymers are that they are easy in processing, high in productivity, and good in cost reduction.

Literature Review

The properties of polymers can be modified by the use of fillers and fibers to get the high strength requirements. Agricultural waste and annual plants can prove to be an alternative raw material for manufacturing of fibrous composite products [1]. Agricultural residue materials like rice and wheat straw are centre of attraction for research works on separation of natural cellulosic fibres by chemical pulping processes. Both the raw materials are renewable and available in abundant quantities in India and other Asian countries. Already some environment friendly and technically compatible processes have evolved and being practiced in industries [2, 3, 4]. The physical properties (specific gravity and moisture content), mechanical property (3-point bending strength), and acoustical property (the sound absorption coefficients) of the composite have been studied to investigate the possibility of rice straw as a partial or complete substitute for wood particles in particleboard. The effects of rice straw size on mechanical properties of particleboards were examined by studying the mechanical properties of particleboards [5]. Pulping processes have been modeled in many ways in order to derive equations for estimating the quality of the resulting pulp as a function of the process variables and researchers have optimized the operating conditions accordingly [6]. The process of improving water resistance of wheat straw based medium density fibreboard (MDF) bonded with amino plastic and phenolic resins have been studied. MDF composites samples meet the ANSI requirement for flexural properties, internal bonding strength and thickness swell test required for interior application [7]. Water absorption behaviour of banana/sisal reinforced hybrid composites has been explored. The tensile, flexural, impact and water absorption tests were carried out for banana/epoxy composite material. Initially, optimum fibre length and weight percentage were determined [8]. In another research, scientists have found that the graphite fibres filled phenolic resin composite exhibited higher flexural strength and flexural modulus than the unfilled composite [9]. The measurement of impact strength showed that the reinforcement of thermosets by 30% (on weight basis) sisal fibres improved the impact strength regardless of the cure cycle used. Curing with first cycle (150 ^{0}C) induced a high diffusion coefficient for water absorption in composites, due to less interaction between the sisal fibres and water [10]. Mechanical characterization (tensile and flexural strength) of woven sisal fabric composites, thermal characterization by TGA and the manufacturing process by compression moulding had been investigated by scientists. Experimental results showed that sisal/phenolic composites have tensile strength and a flexural strength values 25.0 MPa and 11.0 MPa respectively [11]. The physico-chemical characterization of lignin from different sources has been done for use in phenol-formaldehyde resin synthesis. Lignin had been investigated as a promising natural alternative to petrochemicals in phenol-formaldehyde (PF) resin production, due to their structural similarity [12]. A consistent description of temperature modulated differential scanning calorimeter (TMDSC) curves based on irreversible thermodynamics was presented. For time-dependent (linear) processes, the heat flow rate was connected to the convolution integral. This results in a generalized heat capacity which was frequency dependent and, on principle, a complex function of it [13]. TG and DSC analyses had been used to study two aging types, of the polypropylene (PP). The first is natural under solar radiations and rain effects, and the second is artificial by exposition to the light of a 100 W commercial lamp. The thermal degradation is important, more pronounced in the case of the artificial aging than in the natural one, especially for the period of 80 days [14].

Experimental

Materials:

In this research phenol formaldehyde (resole type) and wheat straw fibres were used to manufacture the thermoset composite. Methanol was used as a solvent to dissolve the phenol formaldehyde resin. We have used the chemical pulping process (soda pulping) in lab research digester and intact fibres were used as reinforced materials in composite.

Fibre separation:

To separate the intact fibres from wheat straw we have used 12% NaOH solution. NaOH was used to improve the bonding between reinforcing fibres and matrix (PF resin of resole type). First of all the wheat straw was cut in small pieces (5-7 mm length) and then digested in the pulp digester at 160˚C temperature, 10 kg/cm^2 pressure for one hour of time at maximum temperature. Total time of reaction was two and half hours. Pulp was taken out and washed properly and screened in vibratory slotted screen. Fibres were dried in air until the moisture content was 5-7%. The digester is shown in the figure 1.

Preparation of composites-

The phenol formaldehyde and wheat straw fibres composite was manufactured with the help of semi automatic hydraulic compression moulding machine having the dimension of 107 mm x 150 mm x 3.2 mm. The composites were prepared by the mixture having 40% (weight fraction) wheat fibres and 60% matrix. Properly mixed wheat straw fibres in the matrix was put in the mould and pressed in hydraulic compression moulding machine at different process parameters.

Measurements

1. Tensile strength:

The test of tensile strength was carried out according to ASTM D-638 [15]. The test results are used to evaluate the tensile strength, and tensile modulus from the following equations.

Tensile strength = Force at yield / cross section area

2. Impact strength:

Impact strength test is used to measure the impact force, which may be defined as toughness or ability of material to absorb energy during plastic deformation. The test was carried out according to ASTM D-256 [15].

Impact strength = Energy value taken from pendulum / sample thickness

Figure1: Indirect heating lab research digester

3. Water absorption test

Water absorption behaviour of PF resin and wheat straw composites were carried out at room temperature according to ASTM D-570. All the samples were dried in an oven until constant weight was achieved before being immersed again in the water. The percentage of moisture absorption was plotted against time (hour).

$$\% \text{ of water absorption} = \frac{(m_2 - m_1)}{(m_1)} * 100$$

Where and are initial and final weights of the sample.

4. Chemical resistivity test (alkaline medium)

This test covers the evaluation of plastic materials for resistance to chemical reagents, simulating performance in the potential application environments. The test includes provisions for reporting changes in weight, dimension, appearance and strength properties. Provisions are made for various exposure times, strain conditions and elevated temperature. This test carried out according to the ASTM D-543.

$$\% \text{ of swelling in composite} = \frac{(t_2 - t_1)}{(t_1)} * 100$$

Where and are initial and final thickness of the sample.

5. Differential scanning calorimetric test

With the help of DSC test, the heat flow into or out of the sample is measured while the temperature, at which the sample is exposed, is programmed. When a sample goes through a transition such as from a solid state to a melting stage, or vice versa, heat is either absorbed or emitted without a corresponding change in the temperature of the sample. A small amount of sample, usually 5 to 10 mg is sealed in a conductive pan. Differential scanning calorimetric test was performed according to the ASTM D-7426.

Results And Discussion

Mechanical properties of natural fibres depend on the physical and chemical bond between fibres and

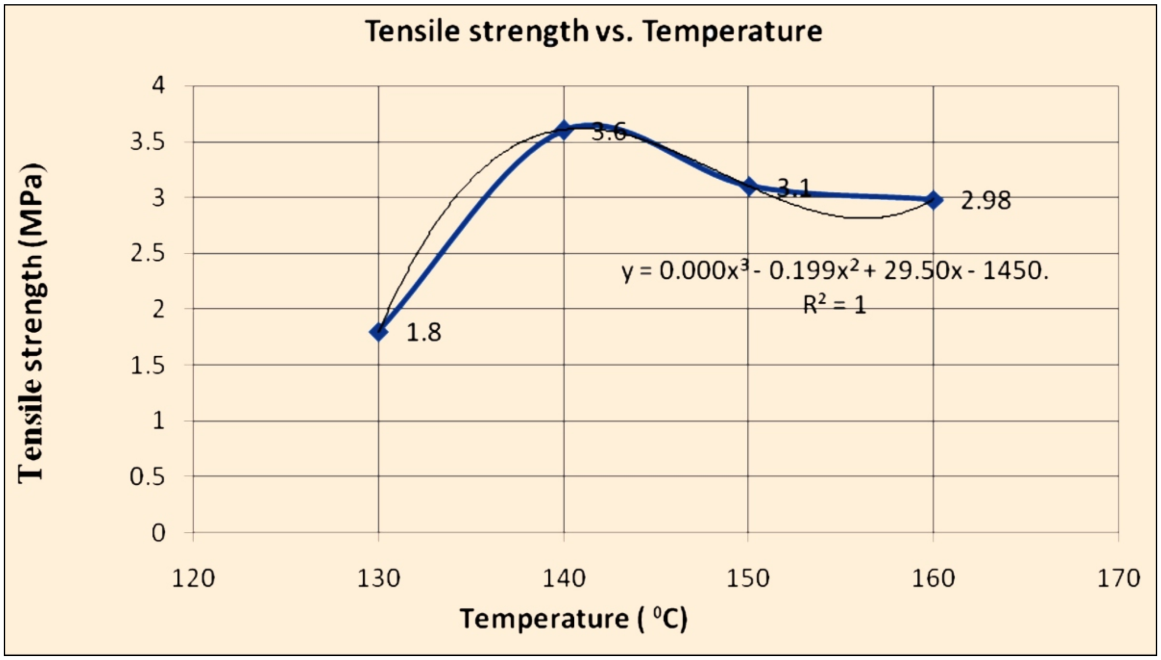

Figure 2: Effect of processing temprature on tensile strength.

matrix. Values of mechanical properties were studied on composite samples manufactured using 40 % (on weight basis) wheat straw fibres in mixture. This shows that the better mechanical properties resulted from the better bonding between the fibres and phenol formaldehyde resin.

In figure 2, temperature was varied from 120 ^{0}C to 160 ^{0}C resulting in increase of tensile strength of composite sample from 1.8 MPa at 120 ^{0}C to 3.60 MPa at 140 ^{0}C and then decreased to 2.98 MPa at 160 ^{0}C. The results showed that maximum tensile strength was obtained at 140 ^{0}C, so this was the optimum temperature for manufacturing the high strength composite sheets. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area.

In figure 3, temperature was varied from 120 ^{0}C to 160 ^{0}C, and impact strength of composite sheets increased from 6.32 kJ/m^2 at 120^{0}C to 12.65 kJ/m^2 at 140 ^{0}C temperature and then decreased to 9.49 kJ/m^2 at 160^{0}C. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area. The results showed that maximum impact strength properties were obtained at 140^{0}C temperature.

As shown in figure 4, in this study temperature was fixed at 140^{0}C and time was varied from 15 minutes to 60 minutes to study the effect of time on the strength properties of composite sheets. Tensile Strength of composite sheets increased from 3.60 MPa at 15 minutes of setting time, to 8.70 MPa at 45 minutes of time, and then decreased to 7.37 MPa at 60 minutes of time. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area.

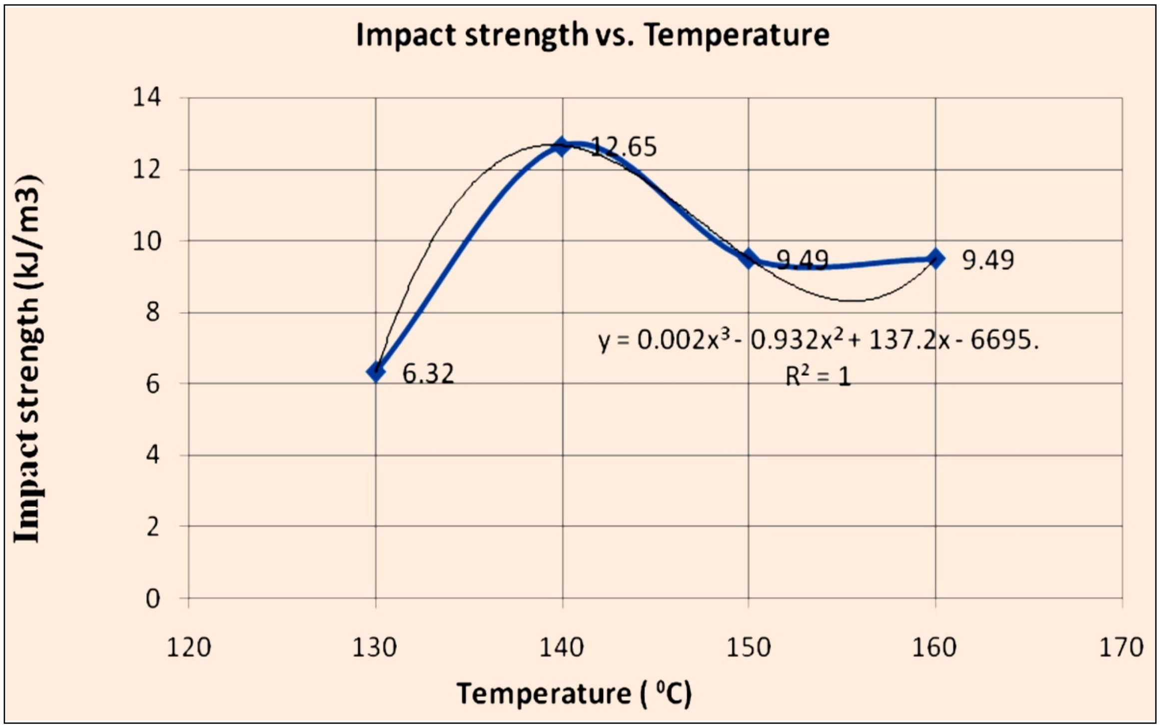

Figure 3: Effect of processing temprature on impact strength

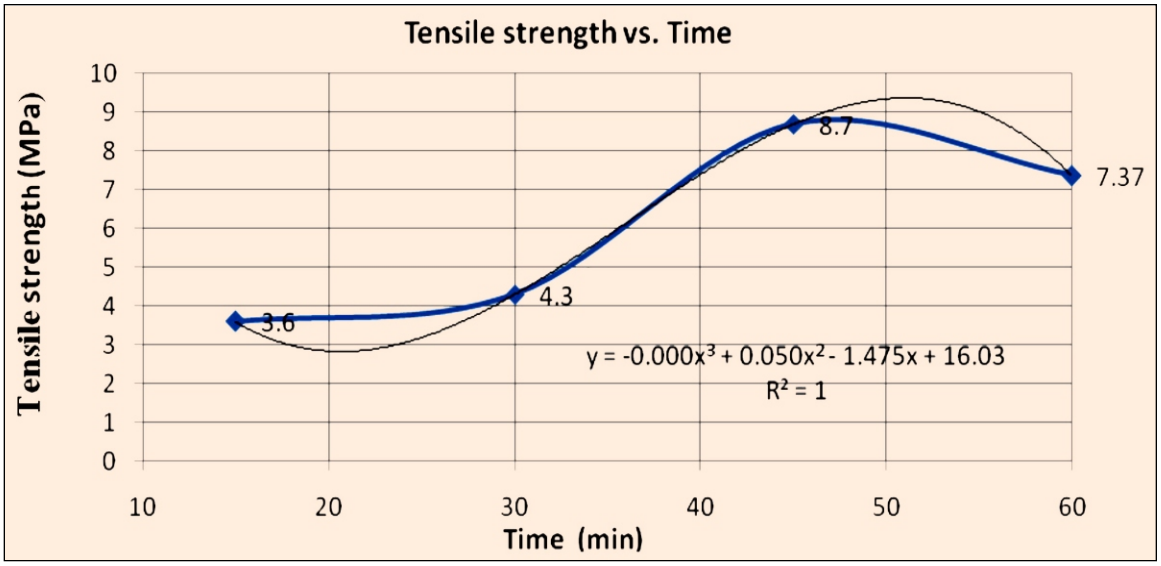

Figure 4: Effect of processing time on tensile strength

In this figure 5, temperature was fixed at 140 ^{0}C and time was varied from 15 minutes to 60 minutes to observe the effect of time on the impact strength properties of composite sheets. The impact strength of the composite sheets increased from 12.65 kJ/m^2 for 15 minutes of setting time, to 21.45 kJ/m^2 for 45 minutes of setting time, and then decreased to 18.38 kJ/m^2 for 60 minutes of time. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area.

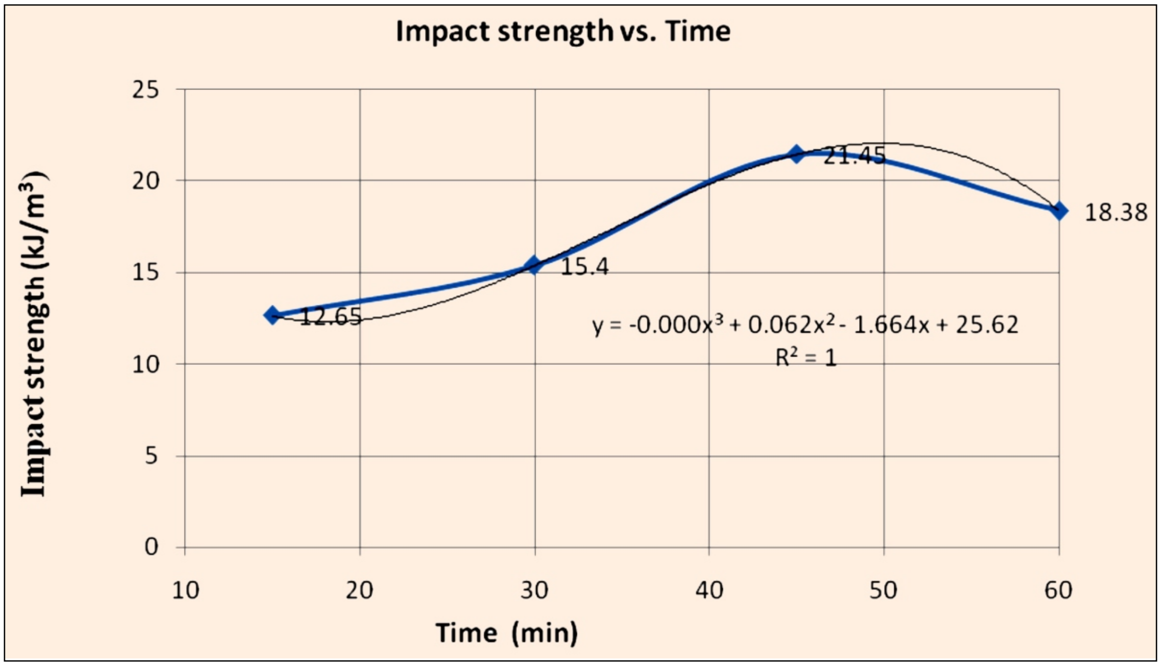

Figure 5: Effect of processing time on impact strength

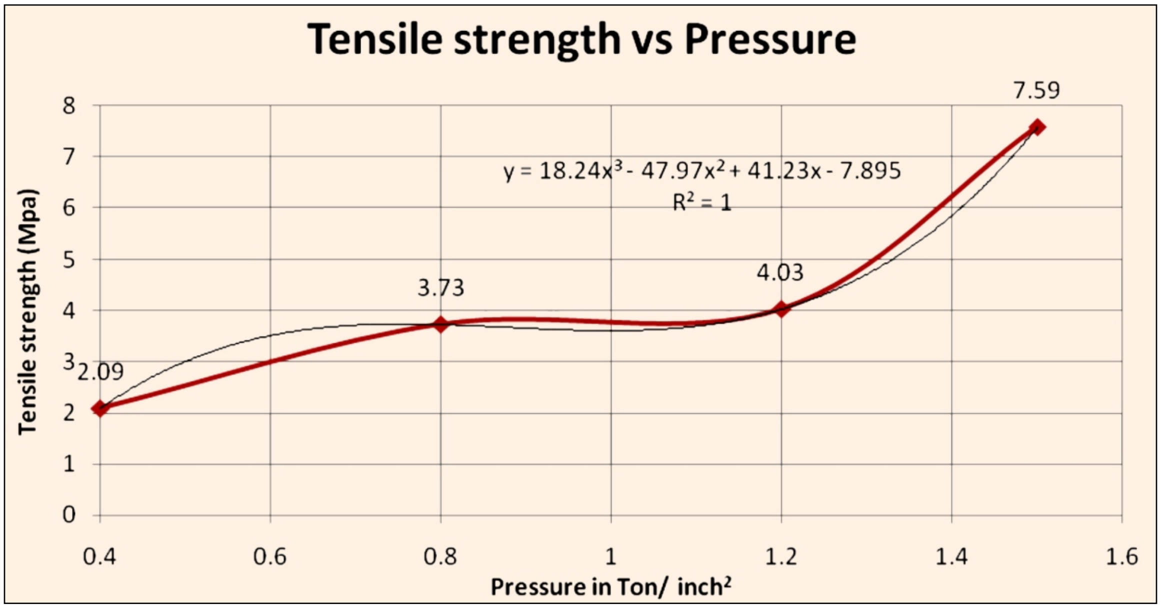

Figure 6: Effect of processing pressure on tensile strength

Now we fixed temperature and time at 140°C, and 45 minutes, and varied pressure from 5.54 MPa to 27.58 MPa to see the effect of pressure on tensile strength of composite sheets. The effect of pressure on tensile strength of composite sheet is shown in figure 6. Tensile Strength increased from 2.09 MPa at pressure of 0.4 tone per square inch (5.54 MPa) to 7.59 MPa at 1.5 tone per square inch (27.58 MPa) of applied pressure in the compression moulding machine. According to the linear experiments, 140°C temperature, 45 minutes of setting time and 27.58 MPa of applied pressure provides the best suitable process conditions for manufacturing composite sheets. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area.

Figure 7 shows that highest value of impact strength

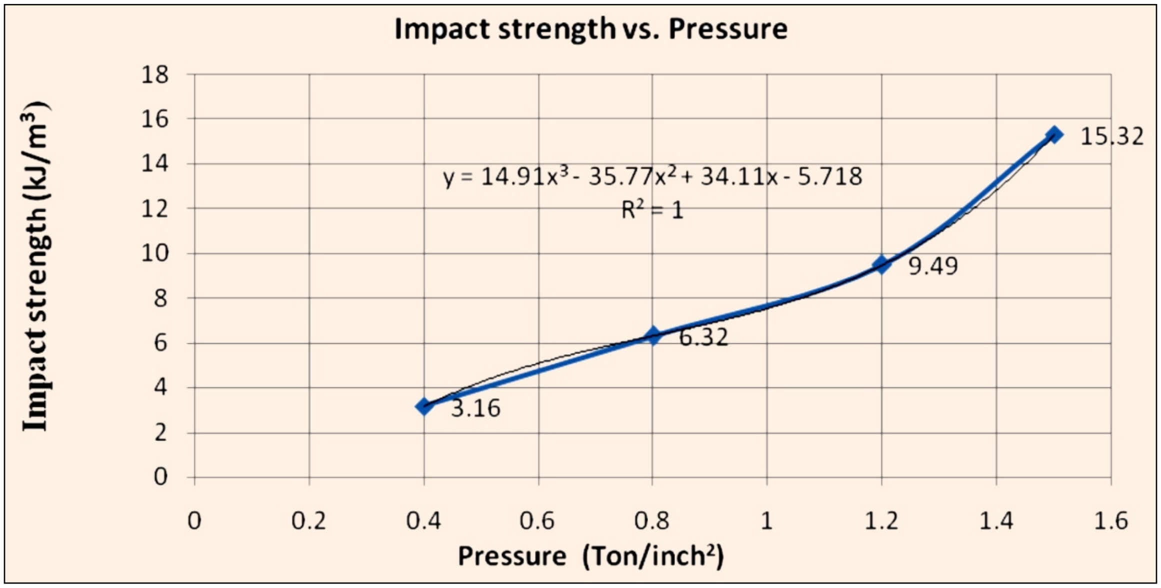

Figure 7: Effect of processing pressure on impact strength

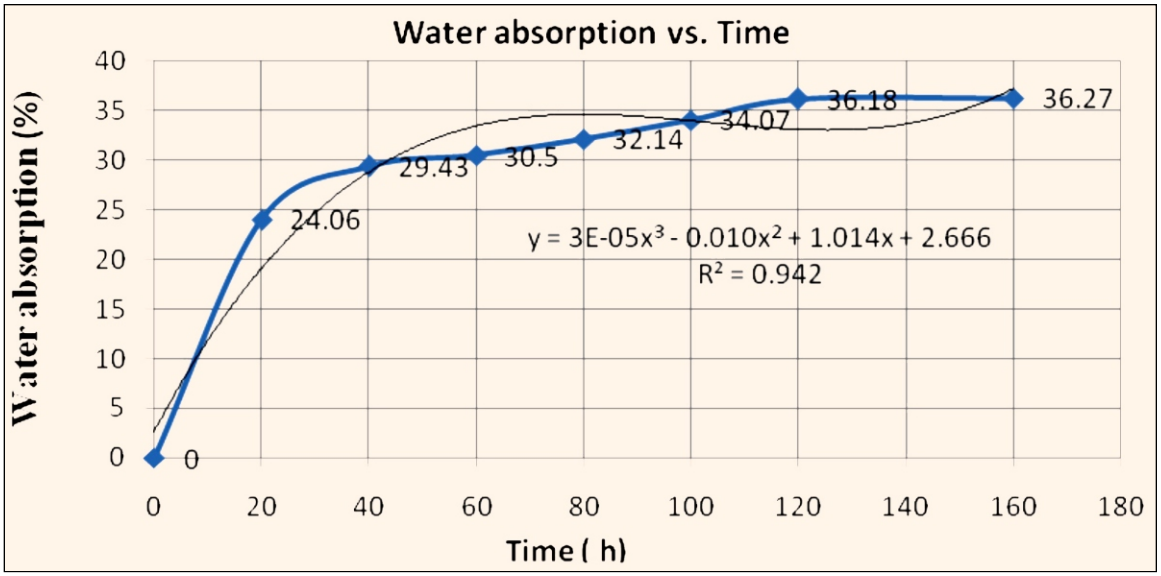

Figure 8: Effect of time on water absorption

15.32 kJ/m^2 was obtained at 1.5 tonne per square inch (27.58 MPa) of applied pressure, 140 ^{0}C and 45 mintue curing Time. Maximum value of Tensile Strength and impact Strength for the samples of manufactured composite of phenol formaldehyde and wheat straw fibres were obtained on 140 ^{0}C temperature, 27.58 MPa pressure, and 45 minute curing time . The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area.

For water absorption test, four samples with the dimension of 10 cm * 1.2 cm* .32 cm were taken and dipped in water for 166 hr in a beaker, and the weight of these samples were measured after every 24 hr. These samples of composite absorbed water from 24% to 36% (weight percentage of water) as shown in figure 8. The black curve shows the trend line for which the curve equation and R^2 values are shown in graph area. These values of water absorption also

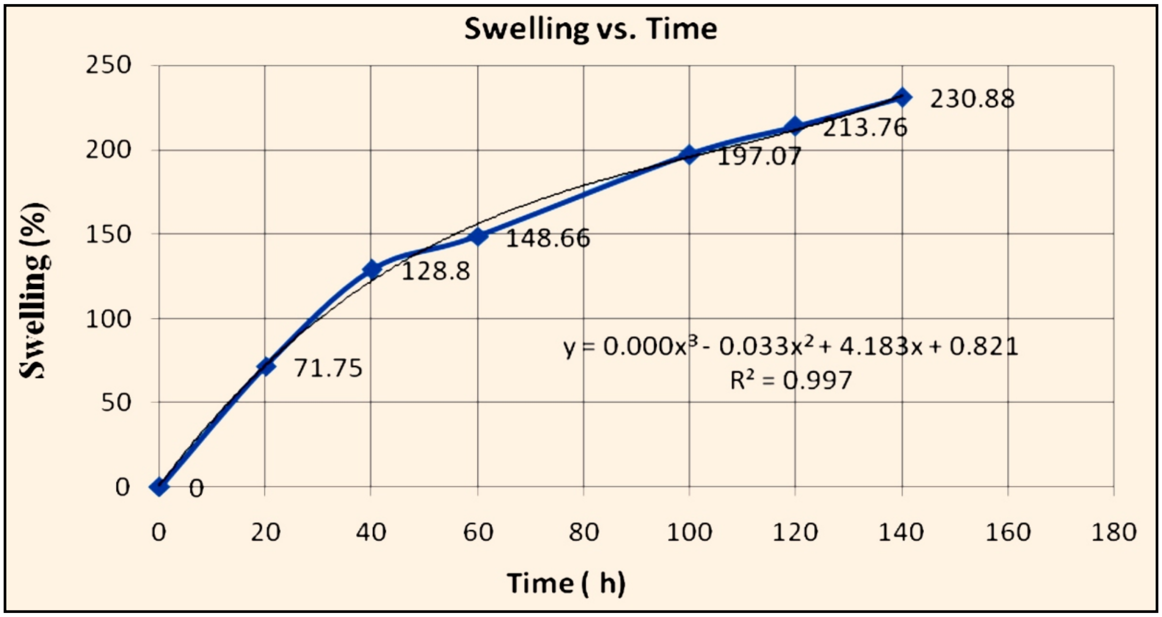

Figure 9: Effect of time on swelling

reflected the good bonding between fibres and matrix. On the other side, this was also shown, that it can be degraded in environmental conditions. Water absorption rate decreased as the time increased, and after a certain time it remained constant, and did not absorbed any more water after 37% (weight percentage) water in composite samples.

Swelling of the test specimens was measured for testing chemical resistivity of composite sheets. The solution of 10 % NaOH was used for measuring the swelling in the composite sheets. The samples were dipped for 167 hrs in 10% solution of NaOH, and the swelling of specimen was measured after every 24 hrs. Alkaline solution reacts very quickly with composite samples, and it was noted that 72 % swelling was observed in the composite samples in first 24 hr as shown in figure 9. The swelling increased continuously with time till the fibres were completely separated from the composite sheets.

DSC test was conducted on the composite samples by heating at constant rate; it showed the endothermic process and first physical change occurred in material at around 108 ^{0}C temperature that could be defined as the glass transition temperature (Tg) of the material. At this point, the sample released some amount of heat and the energy change was measured. After that, temperature of sample further increased and around 120 ^{0}C it was observed as crystalline temperature. This was an exothermic process. Again, the melting temperature of composite was observed at around 131^{0}C temperature and this was an endothermic process. Temperature of 138-145 ^{0}C was observed as the curing temperature of composite; this was an exothermic process as the sample resale some amount of energy. Now the exothermic process continues and temperature of 183 ^{0}C was observed as the degradation temperature and composite material was decomposed after 183 ^{0}C.

Conclusions

The mechanical properties of the composite samples were influenced by temperature, time and applied pressure in the compression mould machine. Wheat straw fibres provided good strength in the composite at 40% weight fraction in the fibre-matrix mixture. The experiments provided the optimum conditions of 140 ^{0}C, 45 minutes of setting time and 27.58 MPa of applied pressure as the best suitable process conditions for manufacturing composite sheets. Water absorption rate was initially high for initial 24 hrs and then decreased as the time progressed further. After a time of 120 hrs, it remained constant and did not absorb any more water. Alkaline medium degrades the composite samples very quickly, and it was noted that 72 % swelling was observed in the composite samples in first 24 hrs. DSC test conducted on the composite samples showed the glass transition temperature (Tg) of material at 108^{0}C, and crystalline temperature 120 ^{0}C. Again, the melting temperature of composite was observed at around 131^{0}C. Temperature of 183 ^{0}C was observed as the degradation temperature of composite material. The composite of wheat straw and PF resin showed good mechanical properties tensile strength 18.76-24.93MPa, impact strength 18.31-25.31 kJ/m^{2} at process conditions of 140 ^{0}C, 27.58 MPa pressure and 45 minute curing time.

Aknowledgement

We want to give special thanks to Dr. Vinay Kumar, Mr. Ramnik Aggarwal, Mr. Naresh Kumar for support in lab works.

Literature Cited

1. Halvarsoon, S., Edlund, H., Magnus, N., *BioResources* 5(2): 1215 (2010).

2. Sinha, A. S. K., *IPPTA* 24 (3): 165(2012).

3. Sinha, A. S. K., Singh, M., Singh, S. P., *IPPTA* 24 (2): 151(2012).

4. Sinha, A. S. K., Singh, M., Singh, S. P., *International Journal of Environmental Engineering* 6 (1): 91(2014).

5. Yang, H., Kim, D., Kim, H., *Bioresource Technology* 86: 117(2003).

6. Jimenez, L., Perez, I., Torre de la, M. J., Lopez,

F., Ariza J., *Bioresource Technology* 72: 283(2000).

7. Hervillard, T., Cao, Q., Laborie, M. P. G., *BioResources* 2 (2): 148(2007).

8. Venkateshwaran, N., ElayaPerumal, A., Alavudeen, A., Thiruchitrambalam, M., *Materials and Design* 32(7): 4017(2011).

9. Wang, S., Adanur, S., Jang, B. Z., *Composites Part B* 28(3): 215(1997).

10. Ramires, E. C., Megiatto, J. D., Gardrat, C., Castellan, A., Frollini, E., *Bioresource Technology* 101(6): 1998(2010).

11. Milanese, A. C., Cioffi, M. O. H., Voorwald, H. J. C., *Composites: Part B* 43(7): 2843(2012).

12. Tejado, A., Pena, C., Labidi, J., Echeverria, J.M., Mondragon, I., *Bioresource Technology* 98(8): 1655(2007).

13. Schawe, J. E. K., Hohne, G. W. H., *Thermochimica Acta* 287(2): 213(1996).

14. Rjeb, M., Labzour, A., Rjeb, A., Sayouri, S., Claire, Y., Perichaud, A., *Physica A* 358: 212(2005).

15. Hylton, D. C., "*Understanding plastic testing*", Hanser Publishers, Munich, Ist Ed. 2004; PP 19, 21, 31, 40.