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RESEARCH ARTICLE

THERMAL, STRUCTURAL, MECHANICAL AND ELECTRICAL PROPERTIES OF BIOMATERIAL
PROSOPIS JULIFLORA

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ABSTRACT

Biomass is the most potential candidate for their quantitative availability. *Prosopis juliflora* a Mesquite is a shrub or small tree in the Fabaceae family. It is also one of the biomass which is available hundred hectares in India. We developed protocol in order to characterize the physical and chemical properties of *P.juliflora* as feed stock for energy conversion process. The particle size of the biomaterial was found to be 8nm from X-Ray Diffraction (XRD) technique. The SEM image reveals the particle size with a cluster of needle shaped surface with voids. Proximate and elemental analyses showed that *P.juliflora* has lower moisture content and high fixed carbon indicates that it is appropriate to meet requirements of thermochemical process. Also, considered as one of the strengths of biomass utilization for energy purposes in terms of contribution to environmental protection, *P.juliflora* contains very low level of Mg and Ca (0.39% & 2.32% respectively). Higher proportion of carbon and lower proportion of oxygen content in *P.juliflora* leads to high calorific value 3891Kcal/kg. Fourier Transform Infrared (FT-IR) was performed for the identification of different modes of functional groups present in the biomaterial. From the microhardness measurements, Vickers hardness number (H_v), Stiffness constant (C_{11}) and Yield strength (σ_y) have been calculated. The electrical behaviour of the biomaterial has been investigated at room temperature. The ac impedance plot indicates a single relaxation process occur at room temperature.

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INTRODUCTION

Biomass constitutes the first energy source human has tamed. Biomass fuels continue to representing the primary source of energy for more than 50% of the world population and amount to about 14% of the total energy global consumption (Kendry, 2002). Amongst different sources of renewable energy, biomass residues hold special promise due to their inherent capability to store solar energy and amenability to subsequent conversion to convenient solid, liquid and gaseous fuels. *Prosopis juliflora* is one of the biomass residue which is used to generate electricity. *P. juliflora* has a negative impact on plant diversity (Singh *et al.*, 2008). The Mesquite tree grows to a height of up to 12 meters (39 ft.) and has a trunk with a diameter of up to 1.2 meters (3.9 ft.). However to use biomass efficiently for energy production a detailed knowledge of its physical and chemical properties are required. These properties more specifically average and variation in elemental compositions is also essential for modeling and analysis of energy conversion process (Nordin, 1994). Ash forming elements such as Si, Ca, Fe, K, Mg, Na, and P in biomass are important to be documented for any thermochemical

conversion process (Cuping *et al.*, 2004). Actually, high contents of alkali are well-known to conduct to critical technical problems when biomass is used as feedstock for thermal power production, since they contribute to slagging, fouling and sintering formation. Actually, information on concentration and speciation of some elements is useful both for energy and environmental issues. Therefore the investigation of physico-chemical properties of biomass fuels would help finding for them suitable and appropriate energy conversion technologies (Obemberger *et al.*, 2004). The importance of hardness has been discussed by various researchers (Nimsha *et al.*, 1997; Pandya *et al.*, 1999; Subhadra *et al.*, 2000; Rao *et al.*, 2002; Sangita *et al.*, 2005) for various applications, since mechanical strength is one of the important properties of any materials represented by its hardness. Measurement of hardness provides useful information about mechanical properties such as elastic constants (Wooster, 1953) and Yield strength (Westbrook 1958). In this paper we report that higher proportion of carbon content and lower proportion of oxygen content leads to high calorific value which is used for planning and control of the combustion plants and also thermal, structural, mechanical and electrical properties of the biomaterial *P. juliflora* are reported.

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MATERIALS AND METHODS

One to two kilograms of *P.juliflora* was collected from the plantation. They were oven dried at 70°C during 24h. The samples were then grinded into powder. The sample prepared with *P.juliflora* powder by pressing it in pellet form with 1.3mm diameter and 1.12mm thickness at a constant pressure of 5 metric tons. The pressed pellet of *P. juliflora* was subjected to the mechanical and electrical studies.

Material characterization

The particle size of the sample was determined using X-ray diffraction (XRD) in a wide range of Bragg angles 2θ ($10^\circ < 2\theta < 90^\circ$) with Co radiation (1.54054 Å). The surface morphology was recorded using scanning electron microscope (JEOL Model JSM-6390LV). The proximate analysis to measure moisture, volatile, fixed carbon and ash content was performed by ordinary oven and muffle furnace (GUNA Model TC141P). The calorific value of the samples was measured using bomb calorimeter. Elements presented in the sample were identified using EDAX (JEOL Model JED-2300) analysis. In order to analyze the presence of functional groups in the sample the Fourier transform infrared (FT-IR) spectrum was recorded in the range of 4000-400 cm^{-1} . Vicker's microhardness measurement is used to study the mechanical properties of *P. juliflora*. For the conductivity studies the pellet was sandwiched between two uniformly polished copper electrodes. The complex impedance spectrum was measured in the frequency range 10Hz-100 KHz using a model SR830 DSP Lock-in Amplifier, Stanford Research Systems, USA. The measurement was performed at room temperature.

RESULTS AND DISCUSSION

Structural analysis

The XRD pattern of the sample shown in Fig.1 reveals the amorphous nature of biomaterial. The size of the sample was determined using Scherr's equation (Vincent 2000) $D = 89 / (1/\lambda \cos \theta)$, where $\lambda = 1.54054\text{Å}$ and $1/\lambda$ is the peak width of the

reflection at half intensity. The average particle size was found to be 8nm. Fig.1 (inset) shows the scanning electron micrograph of the sample at room temperature. Agglomerated and cluster of needle shaped texture was observed.

EDAX Analysis

EDAX analysis of *P.juliflora* shown in Fig.2 has low concentration 0.39% of Mg 2.32% of Ca. The good heat of combustion of *P.juliflorais* due to its higher proportion of carbon and lower proportion of oxygen (Goel *et al.*, 1996). Table1 shows the minor elements presented in the biomaterial.

Table 1. Elemental analysis of *P.juliflora*

Element	<i>P.juliflora</i> Mass%
CK	75.3
OK	17.92
MgK	0.39
CaK	2.32
CuK	4.05

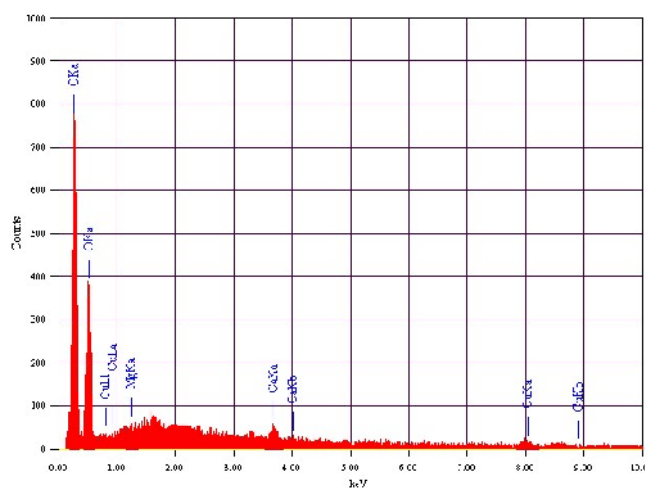


Fig. 2. Elemental analyses of *P.juliflora*

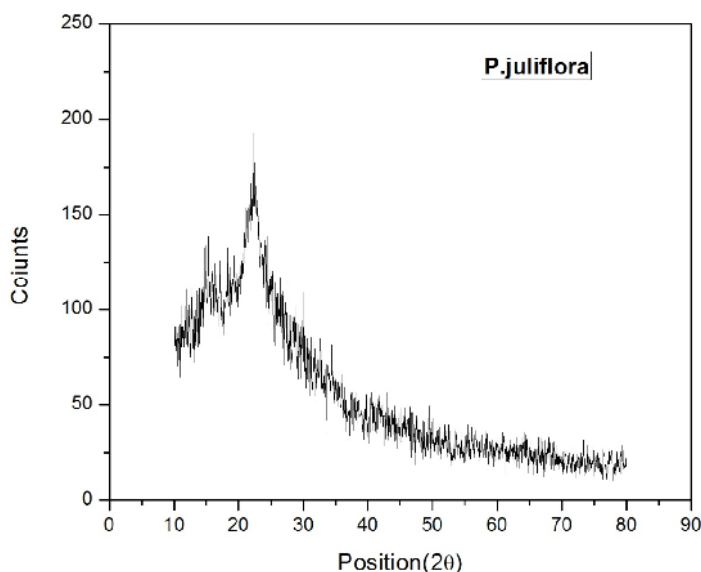


Fig. 1. Room temperature XRD pattern and SEM micrograph of *P.juliflora*

Proximate Analysis

Table 2 shows the result of proximate analysis of *P. juliflora*. Moisture content is of important interest since it corresponds to one of the main criteria for the selection of energy conversion process technology. Thermal conversion technology requires biomass fuels with low moisture content, while those with high moisture content are more appropriate for biological-based process such as fermentation or anaerobic digestion. The moisture content affects the value of biomass as a fuel; the basis on which the moisture content is measured must always be mentioned.

Table 2. Proximate analysis of *P.juliflora*

Moisture (%)	Ash (%)	Volatile matter (%)	Fixed Carbon (%)	Calorific value Kcal/kg
5.35	1.01	79.23	15.69	3891

This is particularly important because biomass materials exhibit a wide range of moisture content (on a wet basis), ranging from less than 10 percent for cereal grain straw up to 50 to 70 percent for forest residues (Jenkins 1980). It is noted from Table 2, that *P.juliflora* has moisture content of lesser than 10% and hence more suitable to serve as feedstock for thermal conversion technologies. The ash content of biomass influences the expenses related to handling and processing to be included in the overall conversion cost. On the other hand, the chemical composition of the ash is a determinant parameter to consider for the operation of a thermal conversion unit, since it gives rise to problems of slagging, fouling, sintering and corrosion. Volatile matter refers to the part of the biomass that is released when the biomass is heated (up to 400 to 500°C). During this heating process the biomass decomposes into volatile gases and solid char. Biomass typically has a high volatile matter content (up to 80 percent), (Jenkins, 1980). Higher proportion of carbon content leads to high calorific value 4200Kcal/kg (Khan *et al.*, 1986). The calorific value is

one of the most important characteristics of a fuel, and it is useful for planning and control of the combustion plants. It indicates the amount of heat that develops from the mass (weight) in its complete combustion with oxygen in a calorimeter standardize.

FT-IR spectral analysis

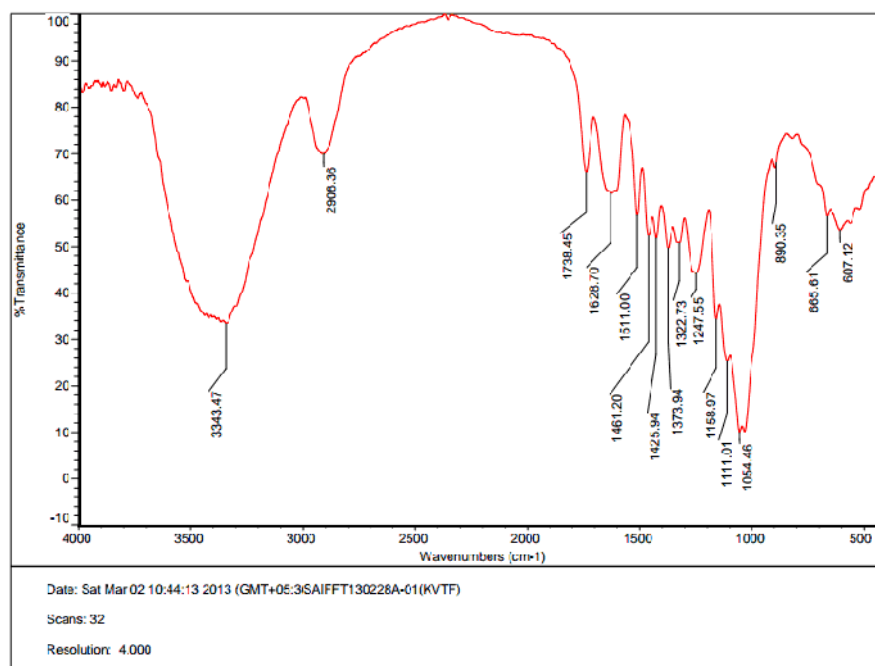
A FT-IR spectrum for the *P.juliflora* is shown in Fig.3. The O-H stretching appears at 3343.47cm⁻¹. The aliphatic (C-H) symmetric stretch appears at 2908.36 cm⁻¹. The C=O stretch appears at 1738.45 cm⁻¹. The peak at 1373.94 cm⁻¹ is assigned for strong CH₂ and CH₃ bend. The region from 1400-600 cm⁻¹ is called finger print region because the pattern of absorptions in this region are unique to any particular compound, just as a person's fingerprints are unique. The observed wavenumbers and their assignments are presented in Table 3.

Table 3. FT-IR spectral Data of *P.juliflora*

Wavenumber cm ⁻¹	Assignments
3343.47	O-H Stretch
2908.36	C-H Stretch
1738.45	C=O Stretch
1461.20	CH ₂ & CH ₃ bend
1373.94	CH ₂ & CH ₃ bend
1247.55	C-O Stretch
1158.97	C-O Stretch
1111.01	C-O Stretch
1054.46	C-O Stretch

Microhardness studies

The Vicker's hardness value was calculated using the relation $H_v = 1.8544P/d^2$ kg/mm² (Pal and Kar 2005) where H_v is the hardness number, 1.8544 is a constant of geometric factor for diagonal pyramid, P is the applied load in kg and d is the diagonal length of the indentation in mm. From straight line graph of log d versus logP (Fig.4), the reciprocal of slope can



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Fig 3. FT-IR spectrum of *P.juliflora*

be obtained and it is equal to the constant n which is equal to Mayer's Index number or work hardening coefficient. The n obtained from the graph is 3.5. According to Onitsch's theory, if n is greater than 1.6 the materials are soft materials (Onitsch 1947). Hence *P.juliflora* belongs to the soft material category. The elastic stiffness constant was calculated by Wooster's empirical relation $C_{11} = H_v^{7/4}$ (Wooster 1953). From the hardness value, yield strength was calculated using the relation $\sigma_y = H_v/3$. The calculated stiffness constant (C_{11}), and yield strength for different loads are tabulated in Table 4.

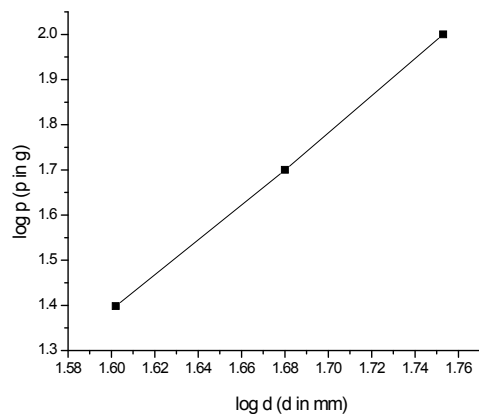


Fig. 4. Graph between $\log P$ vs $\log d$ of *P.juliflora*

Table 4. The elastic stiffness constant for different load

S.No.	Load (g)	$C_{11}(\times 10^{14})\text{Pa}$	YieldStrength σ_y (kg/m ²)
1	25	3.64	9.7
2	50	6.50	13.5
3	100	11.97	19.13

Impedance analysis

The cole-cole plot for *P.juliflora* for frequency range 10Hz-100 kHz at room temperature is shown in Fig.5. The observed semi-circle represents electrical property of the biomaterial. The imaginary component Z'' reaches zero at low frequency, a behaviour indicating that the copper electrodes used were non-blocking type. A non-blocking interface is generally thermodynamically reversible. Equivalent circuit (randles circuit) for the conductivity of the material is shown in Fig.5 (inset).

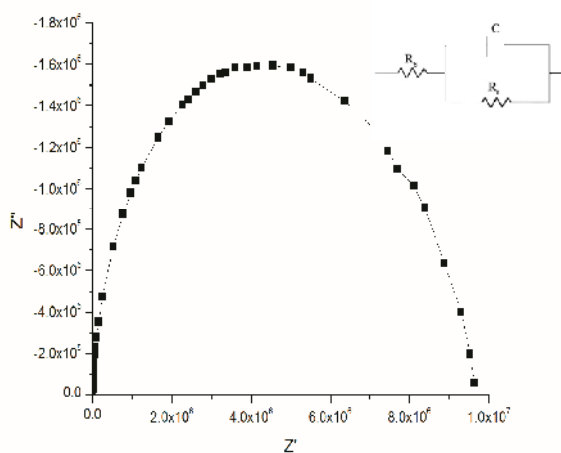


Fig.5 Cole-cole plot of *P.juliflora* at room temperature

Table 5. Relaxation time, Bulk dielectric constant and condition of relaxation

Relaxation time $R_b C_b = 1/\omega_0$	Bulk dielectric constant $\epsilon_b = C_b t/\epsilon_0 A$	$R_b C_b \omega_0$
$6.775 \times 10^{-7}\text{s}$	65.21 F/m	.999

The randles circuit is one of the simplest circuit and most common cell models. It includes a solution of resistance a double layer capacitor and a polarization resistance. The conductivity is derived from the resistance value (R_b) obtained through the semicircles diameter on the intercept with real axis at room temperature using the equation $\sigma_b = t/R_b A$ (Lanfredi et al., 2002). Where t is the thickness, R_b is the bulk resistance and A is the area of cross section of the sample. The conductivity value is calculated as $8.5275 \times 10^{-6}\text{S/m}$. The relaxation frequency (ω_0) at which the complex impedance is the maximum gives the relaxation time. The calculated relaxation time, dielectric constant was shown in Table 5. The calculated value of relaxation time is compared with the papain biomaterial (Muthulakshmi et al., 2013). At the peak, the relaxation is defined by the condition $R_b C_b \omega_0 = 1$ (0.999).

Conclusion

Thermal and EDAX analysis showed that *P.juliflora* is of low moisture content; low proportion of oxygen indicates it is appropriate to meet requirements of thermochemical process. Mechanical hardness studies reveal that *P.juliflora* belongs to soft material. Moderate value of C_{11} indicates that binding forces between the ions are quite strong. Nyquist plot showing the presence of bulk and grain boundary effects in the system. This study could serve to establish a database of biomass fuels or feedstock that would support decision making in terms of energy conversion technology selection and operating conditions setting.

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