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Studies on fatty oil derivatives of Pithcellobrium dulce and Buchanania lanzan: possible industrial application

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Abstract: Amide derivative prepared from the fatty oil of **Pithcellobrium dulce** has been found to disperse calcium and magnesium ions. The property can be used in removing the limitation of fatty acid soaps (usually sodium salt) which are good surface active agents but precipitate in hard water. In the preparation of amide derivative, no solvent has been used, thus a green approach has been attempted. The prepared derivative has been identified by IR, NMR and HPLC. In the second part of the study, preparation of a wood adhesive using natural source – **Buchanania lanzan** flour, has been tried due to high cost and environmental concerns of petroleum based phenol – formaldehyde and urea – formaldehyde resins. The method is economical and ecofriendly. **Pithcellobrium dulce** (Leguminosae)[1] is a cultivated tree, distributed in Central Madhya Pradesh. Two saponins from **P. salman**[2], a triterpenoid glycoside from **P.arboreum** and **P cubense**[3] and chemical composition of **P. multiforum**[4] have been reported. The linoleic –oleic acid rich oil has been analysed in our Laboratory[5] Soaps precipitate with calcium and magnesium, which are present in hard water. To overcome this difficulty, dispersing agents are used alongwith soap based detergents. For preparation of these dispersing agents - large amount of organic solvents are used.[6-8] In the present study, solvent has not been used. **Buchanania lanzan** (Anacardiaceae)[9] is a middle sized tree, distributed in dry hot forests of Bundelkhand[10.] Kernels[11] and bark[12] have been worked out. Fatty oil analysis has been carried in our Laboratory.[13] The flour in presence of phenol and formaldehyde has been evaluated in the present study – to know its performance as a possible adhesive.

EXPERIMENTAL

The oil was saponified using sodium hydroxide - to make sodium soap. It was hydrolysed to yield free fatty acids followed by esterification to give methyl esters. The ester content was subjected to amidation (using diethanolamine and sodium methoxide and heating the contents with stirring).

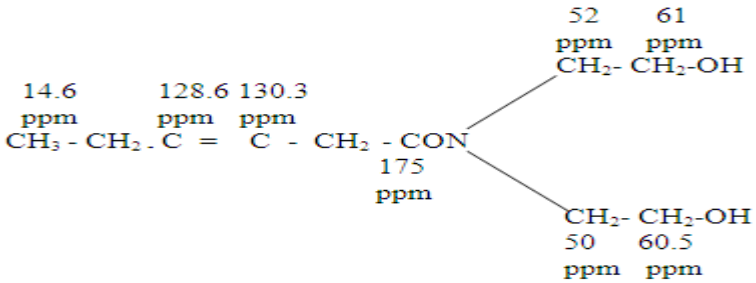
The amide was subjected to IR (KBr pellets), at the range 400 – 4000 cm^{-1}. NMR was recorded on Brucker spectrometer. TMS was taken as standard. Reverse phase HPLC (Waters) was used for further analysis of dieethanolamide.

The Dispersion Power (DP) is determined using 5 ml. of sodium oleate to which is added 10ml. of hard water (containing 600 ppm Ca^{++} and 400 ppm Mg^{++}). This will form soap deposition. Small amount of dispersing agent is added. Test tube is observed to see if there is any dispersion. Minimum amount of dieethanolamide, causing dispersion is noted.

In case of **Buchanania lanzan**, to disrupt the flour – water, sodium hydroxide and ethylene glycol were combined and heated to 70^0C. Now the flour was added to the solution with stirring to form homogenous mixture. The mixture was heated at 70^0C (1 hour) to disrupt the protein content. Formaldehyde was added to modify, disrupted protein with heating at 90^0C (1 hour). This sample was treated with phenol to yield the adhesive. At each stage GPC was performed. For GPC analysis – superpose 12 column with acetonitrile as solvent were used

RESULTS

The proposed derivative (diethanolamide) alongwith its NMR assigenments(Fig.1) can be shown as:



IR spectrum (Fig.2) shows peak for C=O stretching (1620 cm^{-1}) of amide group and C=O stretching (1730 cm^{-1}) of residual methyl ester.

In HPLC, the retention time increases with alkyl chain length. HPLC profile shows peak 1- 4- impurities, 5 – $C_{16:1}$ amide, 6-$C_{18:2}$ amide, 7-$C_{16:0}$ amide, 8 – $C_{18:1}$ amide, 9 – $C_{18:0}$ amide.

The formed derivative is less viscous due to high percentage of linoleic acid amide and is formed in the absence of a solvent. The derivative (amide) is stable to alkaline hydrolysis. The amide –useful as a dispersing agent stops the precipitation of sodium soap. The Dispersing Power (DP) has been found to have the value of 5. From literature [14], effective agents have been found to have DP not exceeding 8. The formed product may find utility for better cleaning and may keep the soap suspended in hard water.

The protein rich flour have amino acid groups which may be available for incorporation into phenol – formaldehyde system. The flour protein after treatement may give a cross linked structure for improved bonding with the surface(wood) . The carbohydrate content of flour may also contribute to stability of adhesive through polymerization. Flour may work out to be econonmical compared to pure protein preparations. To stabilize, the disrupted flour, it was treated with formaldehyde. GPC of formaldehyde added sample (Fig.5) showed high molecular weight peak instead of low molecular ones, due to cross linking. Later it was reacted with phenol (Fig.6). Formaldehyde links protein as well as phenol molecule.

The GPC results (Fig.4-6) were compared at two wavelengths. 220 nm was used to measure flour proteins. Phonolic compounds absorb usually at 273 nm. Absorbtion in this region indicates reactions of flour with phenol and formaldehyde. Flour copolymerizes with phenol formaldehyde, since no free elemental N has been detected. Similarly the product has been termed disrupted and not hydrolysed since no free amino acids have been detected.

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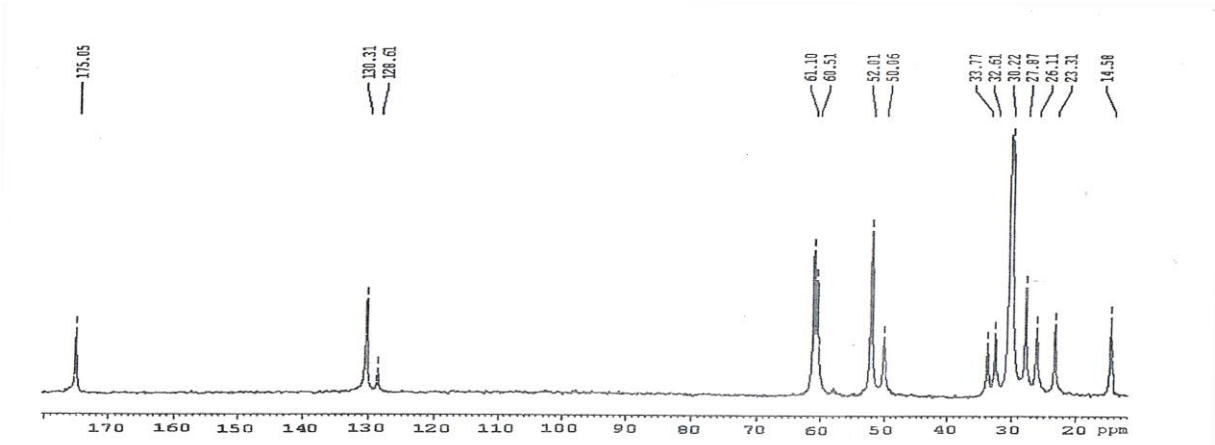

Fig.1: NMR spectrum of the derivative of *P.dulce*

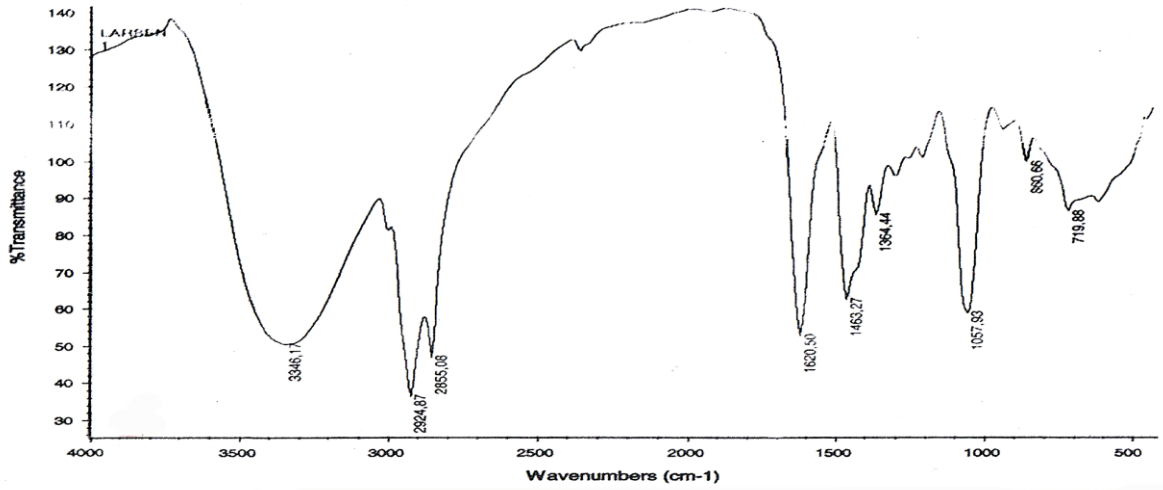

Fig.2: IR spectrum of the derivative of *P.dulce*

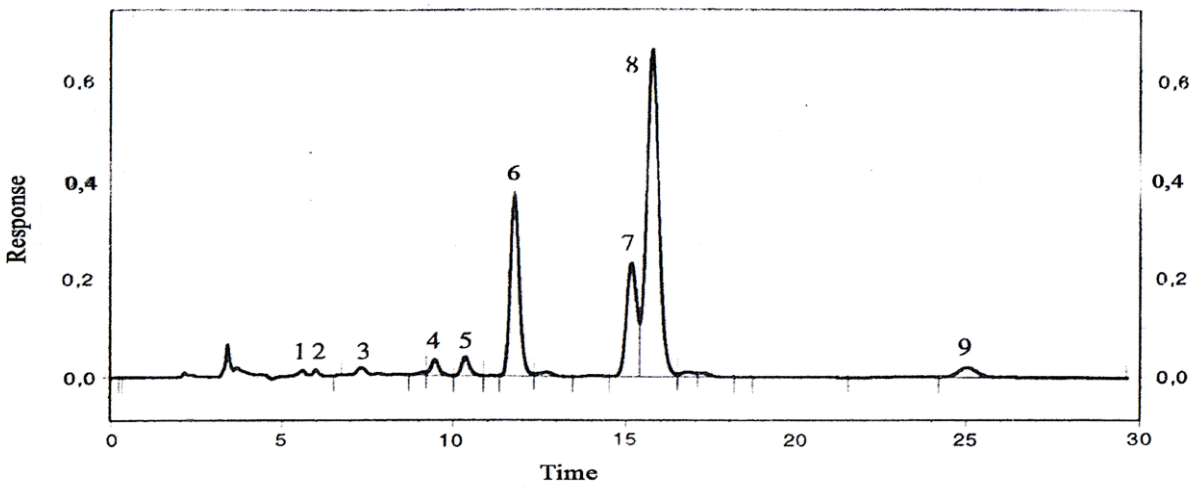

Fig.3: HPLC profile of the derivative of *P.dulce*

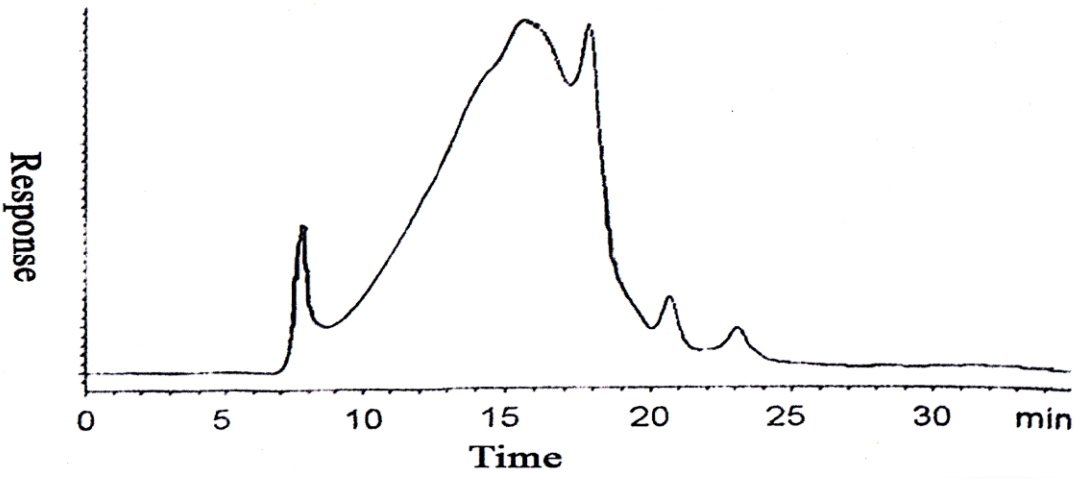

Fig.4 GPC profile of disrupted flour of *B.lanzan*

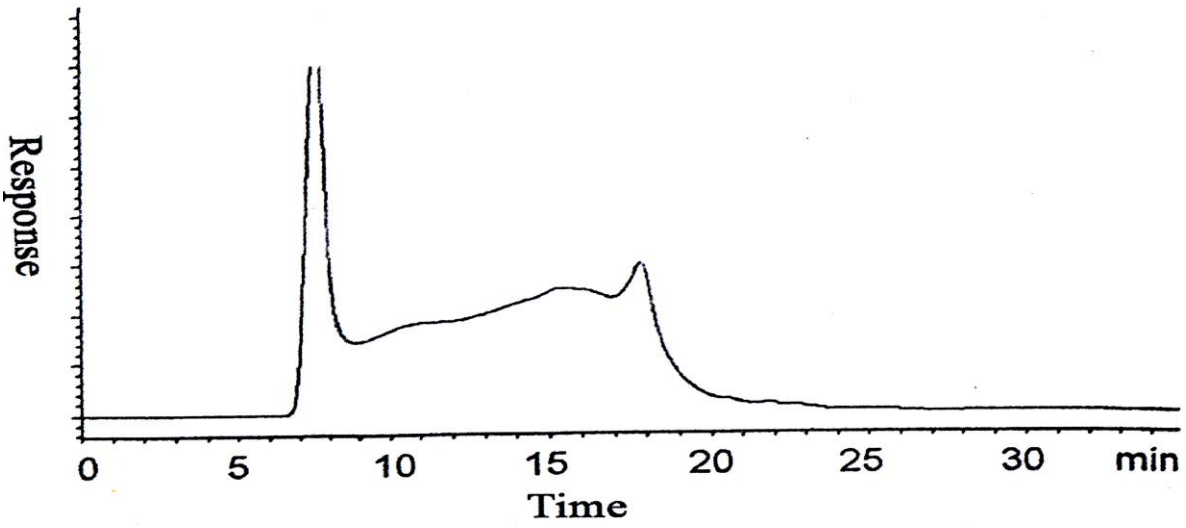

Fig.5 GPC profile of disrupted flour+formaldehyde

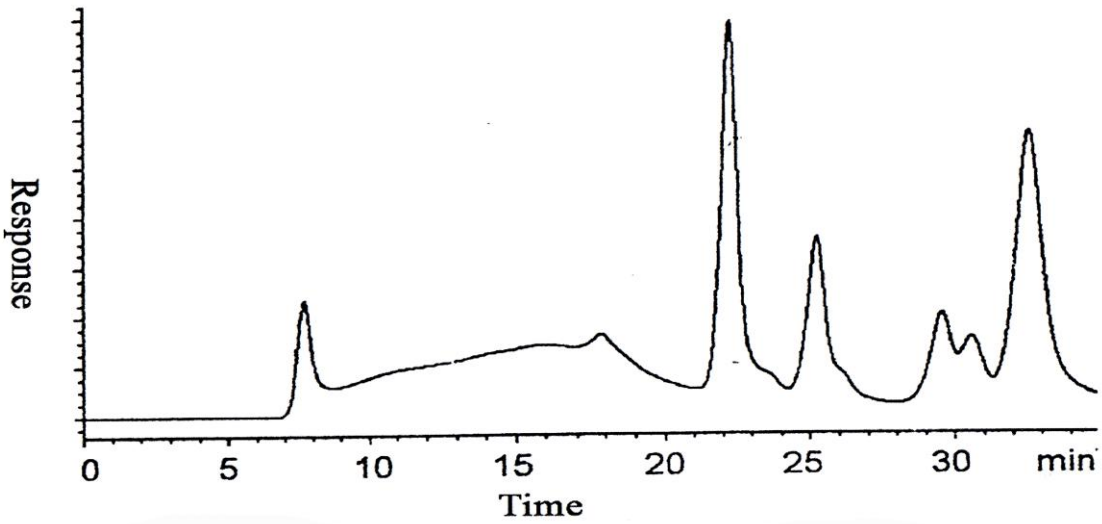

Fig.6 GPC profile of disrupted flour+formaldehyde + phenol