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Synthesis and Characterization of Methylmethacrylate Modified Polyesteramide

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ABSTRACT

In recent years application of renewable resources has become the matter of choice in the field of coating and paint industries. India is an agriculture based country crowned with various plants and herbs. The seed oil of some plants and herbs neither used for edible purpose nor significantly used for medicinal purposes. Polyesteramide resins contain sufficient amide linkages and known to improve water and chemical resistance performances. In the present work Jatropha curcas oil used as a starting material for the synthesis of polyesteramide. The synthesized polyesteramide further modified with methylmethacrylate to improve the afore mentioned performances. Physicochemical characterization of a synthesized resins were carried out as per standard method. The structural elucidation was carried out using IR and NMR dstas.

Key words: *Jatropha curcas,* Seed Oil, Polyesteramide and Modified MMPEA.

INTRODUCTION

In recent years synthesis of the polymers from renewable resources has attracted considerable attention of the research scientists and academia through out the world, because of the continuous hike in price of petroleum and high rate of depletion of natural mineral resources [1-7]. Furthermore, it has also been forecasted that petroleum stocks moving of the point of exhaustion by the late of 21st century [8-10] consequently much attention is focused in the development of materials from forest product which could be grown again and again. This has inspired investigation of the natural renewable sources as an alternative for the polymer industry1, [3, 11-14]. These polymers having great application in different areas such as in adhesive, varnishes, Coatings materials, encapsulating materials and surgical equipments etc [5,6,15-17].

India possess vast forest resources and crowned with various herbs, plant and farm land, yielding variety of oil bearing seeds such as castor[21], linseed[21], Vernonia[21] *Anona squamossa*[22], *Pungamia glabra*[23], soybean[24-25], cocos[26], sunflower[26-27], tung oil[28-29] safflower [29-30], canola [30]etc. The advantage of these polymeric starting materials include their low cost, ready availability and annual renew ability of resultant polymer material after the targeted use [4-5,32]. The oil from various seeds contains fatty acid that vary from 14-22 carbon in length with 1-3 double bond[30,32]. Polyesteramide resins are amide modified alkyds largely used in

paint and Coatings industries because of its better performances against environmental attack.

JATROPHA tree yield non edible seeds which contain about 48% oil into appropriate percentage fatty acid. Although the *Jatropha* oil is of significant importance, but very little work has been reported in literature regarding its utility in synthesizing polymers[31,36]. Furthermore grafting of methylmethacrylate (classified as hard monomer) in polymer chain enhances the hardness of film upto desired lable. Keeping the fact that *Jatropha curcus* will be going to one of the most abundant oil bearing plant.

In present work we have utilized the seed oil of the Jatropha curcas in making polyester amide resin and its modified with grafting of monomer with double objective utilization of a non-conventional seed oil in making coating material and reduce the pressure on utilization of petrochemical

EXPERIMENTAL

Materials

Oil was extracted from cursed seed of Jatropha curcas through soxhlet apparatus, petroleum ether (60-80) was used as solvent. The fatty acids composition of the oil is given in table-1. Diethanolamine purchased from s.d.Fine chemicals, sodium methoxide, xylene and phthalic acid were procured from Merk, India.

Syntheses of N.n-bis (2-hydroxy Ethyl) *Jatropha curcas* Oil Fatty Amide (Hejca)

Diethanolamine 0.32 mole and sodium methoxide 0.007 mole were taken in four neck round bottom flask fitted with an electrical stirrer, thermometer, dropping funnel and condenser. The reaction mixture was heated to 120-140^{o} C . The Jatropha curcas oil (0.1 mole) was added drop wise into the reaction mixture over a period of one hour. The progress of reaction was monitored by TLC[35].

After the completion of reaction the product was dissolved in diethylether and washed with dilute aqueous 15% sodium chloride. The etherial solution was filtered and evaporated in a rotatory vacuum evaporator to obtain HEJCA.

Synthesis of *Jatropha curcas* Polyesteramide (Jcpea)

HEJCA, pthalic acid in equal molar ratio and xylene as a solvent were placed in four necked round bottom flask fitted with a Dean Stark Trap, thermometer and mechanical stirrer. Reaction mixture was heated up to 160^{o} C. The progress of reaction was monitored by taking acid value at regular interval.

Characterization

The chemical characterization acid value, iodine value, hydroxyl value and saponification value of the oil HEJCA, Polyesteramide and MMPEA was done as per standard laboratory method and given in Table 1. The solubility of polyesteramide and MMPEA in various organic solvent was checked at room temperature.

Preparation and Testing of Polymeric Coatings

Coating of JCPEA and MMPEA were prepared on commercially mild strip 30x10x1 mm for chemical resistance and 70x25x1 for scratch, hardness and impact resistance. Coated samples were baked for 5-35 minutes in an oven at different temperatures (140-160^{o} C) to find out the optimum baking time and temperature. The best coatings were obtained by baking at 150°C for 20 minutes. The coating thickness were found between 120-150 µm.

RESULTS AND DISCUSSION

Chemical reaction and polymerization scheme of HEJCA , JCPEA MMPEA are given in Fig. 1.The progressively decrease in acid value confirm the formation of polyesteramide

The various physical and chemical characterization of the oil, HEJCA and JCPEA are given table 1 and table 2.Table 2 shows progressive decrease in iodine value confirm the increase in chain length of the polymer.The performances of the coating material in different chemical and corrosive environment is summarized in table-3.It is found that stability of JCPEA coatings is quite good in saline and acidic environment, however the performances in alkaline solution is poor. The JCPEA coating passes the flexibility test on 1/8 and 1/4 conical mandrels. The scratch resistance performances of the coating materials was studied

Table 1: Characterization of *Jatropha curcas* seed oil

Characteristic	*Jatropha curcas* oil
Oil content	40%
Gardener color (no.)	4.0
Specific gravity	0.927
Refractive index	1.475
Iodine Value	75.18
Acid Value	11
Saponification Value	180

Table 2: Characterization of HEJCA and JCPEA

Characteristic	HEJCA	JCPEA
Yield	80.68	85.16%
Acid Value	8	10.6
Iodine Value	30	24
Saponificaton Value	144	132
Specific gravity	0.938	0.948

Table 3: The Physico-mechanical properties of JCPEA

Test		Performance
1.	Flexibility (1/8, 1/4 Conical Mandrals)	Pass
2.	Scratch hardness (in kg)	2.5
3.	Immergence test	
	a. 2% HCl Solution (60 h)	A
	b. 2% H_2So_4 Solution (48 h)	B
	c. 3.5% NaCl Solution (2 h)	B
	d. 5% NaOH Solution (2 h)	D

A= unaffected, B= slight loss in gloss, C= slight loss in gloss and weight and D= fill ruptured

on scrath harder apparatus. The coating of JCPEA passes the scratch hardness test upto the 2.5 kg. These results are comparable with other oil based coatings.

CONCLUSION

The polyesteramide of Jatropha curcas seed oil is found to comparable with the reported traditional oil based polyesteramide. The color value of Jatropha curcas polyesteramide is very low therefore it provides an additional opportunity to develop different coloured coating materials. The JCPEA holds promise for commercial application.

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