

## **FINAL REPORT**

**PROJECT TITLE:** “Preparation and Characterization of Binders (Styrenated and Vinylated) for Surface Coating Applications”.

**PROJECT NO:** 47 -1989/11](WRO) dated 22<sup>nd</sup> February 2012

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## **Final Report of the work done:**

### **1 INTRODUCTION:**

Salicornia oil was first converted into N,N-bis-(2-hydroxyethyl)Salicornia fattyamide (HESFA) by reacting salicornia oil with diethanolamine using zinc oxide as a catalyst. Prepared HESFA was reacted with different amount of phthalic anhydride to obtain salicornia oil based polyesteramides (SPEAs) .

The prepared SPEAs were then reacted with varying ratio of styrene and vinyl acetate. The resins were tested for chemical properties like acid resistance ,alkali resistance Xylene resistance and for mechanical properties.

### **2. EXPERIMENTAL**

#### **2.1 Materials:**

Salicornia, Diethanol amine, ether, phthalic anhydride toluene ,styrene, Vinyl acetate.and all other chemicals used for the study were of laboratory grade.

#### **2.2.1 Preparation of N,N –bis (2-hydroxyethyl)Salicornia fatty amide.[HESFA]:**

Salicornia oil (1.0 mole) was heated with diethanolamine (3.0 moles) under constant stirring at 200 °C for 15 minutes, using zinc oxide (0.02 moles) as a catalyst and HESFA was synthesized as per the reported method. Nitrogen was used to create inert atmosphere. Thin-layer chromatography (TLC) method was used to check the completion of reaction.

To wash HESFA and remove the impurities diethyl ether and 15 percent aqueous sodium chloride solution followed by distilled water was used.Glycerol, excess diethanolamine and other water soluble impurities was removed.

#### **2.2.2 Preparation of Salicornia Oil Polyesteramides [SPEAs ]from HESFA:**

Polyesteramides were prepared by reacting HESFA (1.25 mole, 1.40 mole) with phthalic anhydride (1.0 mole) as per the reported method.Under the blanket of nitrogen the reactants were heated slowly with constant stirring at the temperature of  $175 \pm 5^{\circ}$  C for 3.5 hrs. Azeotropic distillation was used to remove water of the reaction .Xylene was used as a solvent in a Dean- Stark moisture trap. The

samples were checked for acid value at regular interval of time to study the progress of the reaction. The reaction was allowed until low acid values were obtained.

### **2.2.3 Styrenation and Vinylation of Polyesteramide Resins)**

Poststyrenation of the polyesteramide resins was carried out using benzoyl peroxide as a catalyst. Different PEA/ Styrene+ Vinyl acetate ratios were prepared and reacted with polyesteramides.

Mixture of Polyesteyramide resin (60% in xylene, 133 parts) was charged into a multi- necked flask, fitted with an efficient condenser, a mechanical stirrer, a thermometer and a means for purging dry nitrogen gas. With continuous reflux reaction mixture was heated and brought at 140<sup>0</sup> C. Using a separating funnel, Styrene and Vinyl acetate were added to the reaction flask slowly over a period of 4 hrs. In styrene Benzoyl peroxide (2.5% by wt of polyesteramide) was previously dissolved. The reaction mass was stirred for further 4 hrs at 140 ± 5<sup>0</sup> C after the addition was complete. Periodically the samples were withdrawn from the reaction flask and checked for viscosity and clarity. When no significant increase in viscosity was observed between two consecutive samples the reaction was stopped. The prepared resin was then thinned with xylene to 60% nonvolatile content.

## **3.0 RESULTS AND DISCUSSION:**

### **3.1 Spectral Characteristics of Polyesteramide of Salicornia:**

The strong absorption IR band at 3400 cm<sup>-1</sup> suggests the presence of residual hydroxyl group in the polyesteramide of salicornia .A strong band at 2854 cm<sup>-1</sup> is due to the C-H stretching of the methylene group present in the fatty acid .The estercarbonyls present shows a strong absorption band at 1738cm<sup>-1</sup>.

The C-H stretching and amide carbonyl group presence shows a weak band at 1651cm<sup>-1</sup> respectively. The strong band at 780 cm<sup>-1</sup> and weak band at 790 cm<sup>-1</sup> and 1070 cm<sup>-1</sup> each can be attributed to the aromatic moiety present in the structure .

### **3.2 Styrenated Vinylated Salicornia oil based Polyesteramides Characteristics:**

Polyesteramides and styrenated vinylated polyesteramide resins were compared for different chemical, mechanical and anticorrosive properties. The styrenated polyesteramide's viscosity and clarity was higher than that of the polyesteramides. The result indicates that with decrease in PEA/ Styrene Vinyl ratio, viscosities of resins increases and degree of styrenation increases. The result also indicates that the high degree of polymerization takes place when equal amount of styrene and vinyl acetate is used.

The PEAs have dark color compare to their styrenated vinylated resins. The color decreases with decrease in PEA/styrene vinyl ratio.

### **3.3 Film Properties of Styrenated Vinylated Salicornia oil based Polyesteramides**

The surface-drying (dry-to-touch) time, of styrenated and vinylated polyesteramide reduces significantly in all the families of the resins. As PEA/Styrene Vinylation ratio decreases the drying time decreases. This is due to high strength provided by the polystyrene moiety in the resin structure. Styrene to vinyl ratio 2:1 gives better quality resin with reduced drying time .

Air dried and baked panels were subjected to the flexibility test on ¼” conical mandrel and test for adhesion. All the samples of the baked film passed the test for both. Impact resistance and Scratch hardness increases with decrease in PEA / Styrene Vinyl ratio. It was also observed that styrenated and vinylated polyesteramide showed improved resistance to impact and greater scratch hardness than polyesteramides. The baked films have better properties than air dried resins. The polystyrene moiety gives better scratch hardness of the air dried and baked films of all the families of the resins showed an improvement with decrease in PEA/Styrene ratio. The value of impact resistance of air dried films are comparable but impact resistance decreases when PEA/Styrene Vinyl ratio was 2:1 in baked films due to film embrittlement.

The scratch hardness and impact resistance of styrenated polyesteramide and polyesteramide is better compared to their PEAs.

Styrenation and vinylation of polyesteramides had improved water, acid and alkali resistance properties in both the air dried and baked films. Properties like acid,

alkali and water resistance were improved with decrease in the PEA/Styrene ratio. Acid, alkali and water resistance of baked films is superior to their air dried films. Due to increase in styrene content, there is corresponding decrease in hydrolysable ester linkages in the matrix of the film which results in improved alkali, acid and water resistant. The xylene resistance decreases with the increase in styrene content as xylene an organic solvent easily attacks the film of prepared resin.

#### **4. CONCLUSION :**

PEAs can be styrenated and vinylated easily by post styrenation technique using benzoyl peroxide as a catalyst. The improved film properties can be obtained using styrenation and vinylation of polyesteramides. Baked film properties of the are superior than air dried films. The improved chemical, water resistance and scratch hardness except xylene resistance were observed.

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