

Development Studies of Biodegradable Pressure Sensitive Adhesives and Fiber Reinforced Polyester Composites from Castor Oil

J. Shakina^{1*} and A. Muthuvinothini¹

¹Department of Chemistry, Sarah Tucker College (Autonomous), Tirunelveli, Tamil Nadu 627007, India.

Authors' contributions

This work was carried out in collaboration between both authors. Authors JS designed the study and wrote the protocol. Author AM performed the statistical analysis, managed the literature search and wrote the first draft of the manuscript with assistance from author JS. Both authors read and approved the final manuscript.

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ABSTRACT

The potential use of castor oil in preparation of novel biodegradable polymeric pressure sensitive adhesives and fiber reinforced polyester composites was investigated. A biodegradable oligomeric fumarated resin was prepared by the insitu hydroxylation of castor oil followed by maleation under controlled reaction conditions. Then two varieties of biodegradable pressure sensitive adhesives (PSAs) were prepared by further reacting the resin with co-monomers vinyl acetate and methyl acrylate. The PSAs were characterized for biological and physicochemical properties; namely thermal stability, transparency, chemical resistance, biodegradability, and tensile strength. Fiber reinforced polyester composites were prepared by free radical polymerization of oligomeric fumarated resin mixed with different proportions of Jute and Sisal fiber. The composites were then analyzed for physicochemical by AT-IR, SEM, TGA and DSC techniques. The results indicate that

*Corresponding author: E-mail: shakinajudson@gmail.com;

castor oil-based PSA films and tapes have applications in various fields including flexible electronics and medical devices because of their thermal stability, transparency, chemical resistance, and potential biodegradability from triglycerides. Mechanical characterization of the composites was also done and the results positively indicated the Sisal and Jute fibers have the potential to be used as substitutes for synthetic fibers in green composites. The composites can be used for the fabrication of eco-friendly materials.

Keywords: Pressure sensitive adhesives; methyl acrylate; biodegradable; Jute; sisal; polyester composite.

1. INTRODUCTION

Polymers have extensive application in the aerospace, automotive, marine, infrastructure, military, sports and other industrial fields. These lightweight materials exhibit excellent mechanical properties, high corrosion resistance, dimensional stability and low assembly costs. Traditionally, polymers and polymeric composites have been derived from petroleum derivatives; however, as the application for polymeric materials increase, finding alternative sources of these materials has become critical [1]. The increase in polymeric material demand has partially contributed to increases the price of petroleum-based polymeric materials, which in turn has heightened awareness of renewable alternatives for polymeric feed stocks. Cellulose, starch, proteins and natural oils have all been examined as possible polymeric feed stocks [2]. The application of natural fibers as alternatives to synthetic fibers in polymer-fiber composites has similarly generated great interest. In recent years, a broad range of chemical routes were developed to use natural triglyceride oils to make polymers and polymer composite materials [3]. These materials have economic and environmental advantages that make them attractive alternatives to petroleum based materials. Among others, those advantages include biodegradability, low toxicity, high purity and ready availability [4].

Pressure sensitive adhesives (PSAs) are viscoelastic materials with flow properties playing a key role in the wetting and debonding process. These materials present an interesting class of products that offers a great potential to produce a novel generation of self-adhesives with numerous excellent properties. The main advantage of the PSAs compared with other types of adhesive is their convenience of use, such as no storage problem and the often readily reversible bond [5]. Similarly, the potential of producing newer composites using alternative reinforcing phases continue to be a subject of serious investigation [6-10]. According to

Morassi, sisal fiber, considered as an alternative to glass fiber, can be satisfactorily used as a reinforcing agent in plastic products for many applications in the automobile industry [11]. Rao et al. investigated the possibility of introducing natural fibers into a polymeric matrix for production of economic and lightweight load-carrying composite structures [12]. Dick et al. conducted static and cyclic 4-point bending tests on glass-filled polycarbonate, to collect results for evaluation of a theoretical model on its capability to predict the fatigue life and the residual strength after the cyclic loading [13]. Wang et al. investigated the effective thermal conductivity enhancement of carbon fiber composites using a three-dimensional numerical method [14].

The evaluation of sisal fiber reinforced polyester composite from castor oil has been a much less studied area. A lot of research has been done on natural fiber reinforced polyester composites but research on jute based polymer composites is very rare [15]. So, the present research work has been undertaken with objectives to synthesize pressure sensitive adhesives and sisal and jute fiber reinforced polyester composites using oligomeric fumarated resin from castor oil and to study their properties.

2. MATERIALS AND METHODS

Castor oil purchased from the market was used as received. Sisal and jute fiber were obtained from Sarah Tucker College campus, Tirunelveli-7, India. Vinyl acetate (VA), Methyl acrylate (MA), Formic acid (98%), Hydrogen peroxide (30%) and Styrene were purchased from Merck Chemicals. Benzoyl peroxide, Dimethyl aniline (DMA) and Morpholine (MP) were purchased from Aldrich Chemicals Pvt. Ltd. All the above chemicals were used without further purification.

2.1 Synthesis of O-PTF Resins

Triglyceride castor oil was mixed with of 97% formic acid and 550 ml 30% (H₂O₂). Ice water was used externally to keep the temperature

below 40°C. The reaction was vigorously stirred overnight. The resulting emulsion was poured into a separating funnel and extracted with ether. The aqueous layer was washed with water, dilute Na₂CO₃ and saturated NaCl solution. The resulting ether layer was dried over anhydrous sodium sulphate and the ether was removed by a rotatory evaporator. The resulting product was hydroxylated triglyceride oil resin (HTO). The hydroxylated triglyceride resin was then reacted with maleic anhydride to get oligomeric polymeric triglyceride fumarate resin (O-PTF resin). An amount of 2 ml of Morpholine was used as catalyst. The synthesis of fumarated triglyceride castor oil is explained in Fig. 1.

2.2 Synthesis of Pressure Sensitive Adhesives (PSAs)

The two PSAs were prepared by free radical polymerisation of O-PTF resin of castor oil with cross linking co-monomers, VA and MA, at room temperature with benzoyl peroxide as free radical indicator and dimethyl aniline as accelerator. To prevent oxygen free radical reaction, the resin was purged with nitrogen gas prior to curing and then cast on a clean glass plate covered with silicone oil and then left to cure for 6 hours at room temperature.

2.2.1 Synthesis of PSA by adding methyl acrylate and vinyl acetate

Pressure sensitive adhesives were prepared by reacting fumarated triglyceride castor oil with co-

monomers vinyl acetate and methyl acrylate and labeled as PSA-1 and PSA-2. Fig. 2 shows the scheme of the synthesis of PSA-1 using vinyl acetate co-monomer. The above scheme was the same for the synthesis of PSA-2 when methyl acrylate was used as the co-monomer.

2.3 Synthesis of castor oil Resin-fiber Reinforced Biopolyesters (CSFRP and CJFRP)

Fiber (jute and sisal) were washed thoroughly in a 0.1M NaOH solution followed by water washing, air drying, and cutting in to short fiber form of 3 mm of length. The fiber reinforced polymer was prepared by free radical polymerization of (O-PTF) resin with the cross linking co-monomer styrene and approximate composition (5, 10, 20, 30, 40%) of fiber at room temp with benzoyl peroxide as free radical initiator and dimethyl aniline as accelerator using compression moulding process. To prevent oxygen free radical reaction, the resin was purged with N₂ gas prior to curing, and then cast on a clean glass plate covered with silicone oil, and then cured for 6 hours at room temperature.

2.4 Analysis of Triglyceride Oil and (O-PTF) Resin

Castor oil (Refined), hydroxylated resin and fumarated resin were analyzed for specific gravity and characterized using UV-Vis and FT-IR analysis.

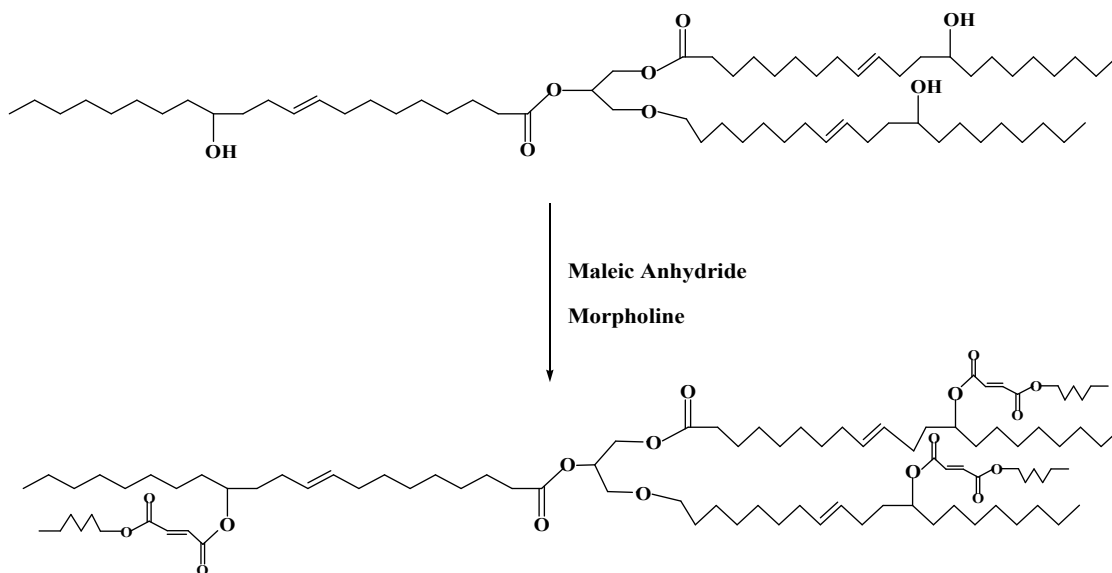


Fig. 1. Structure of fumarated triglyceride castor oil

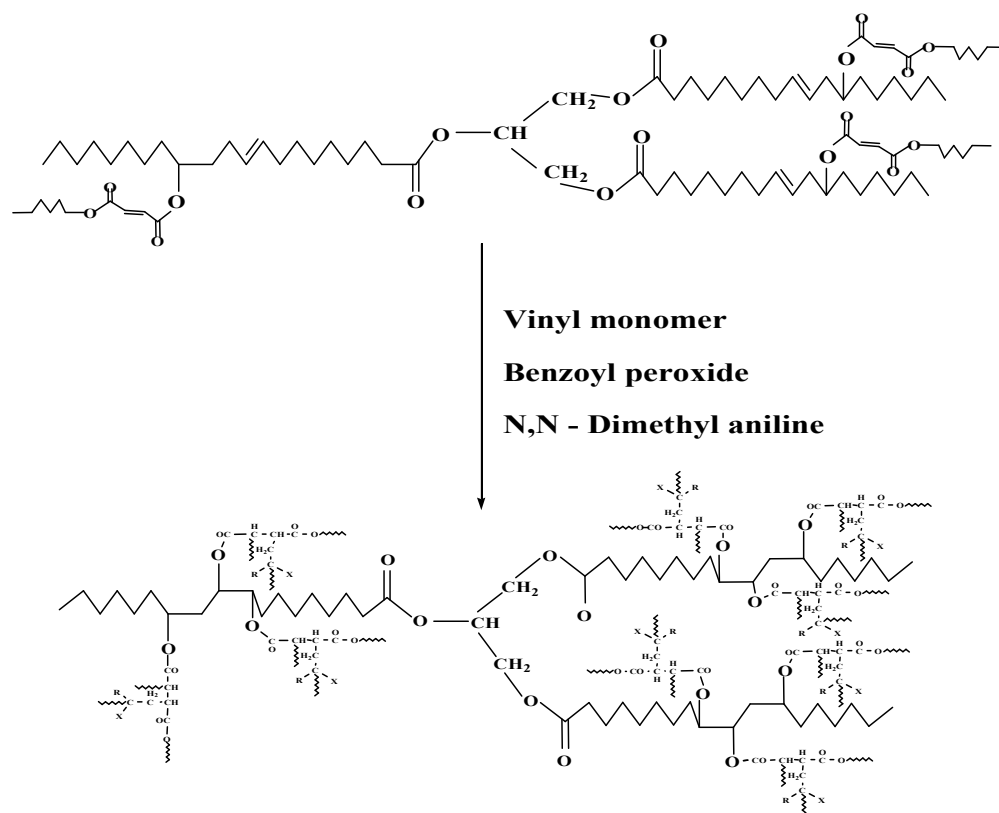


Fig. 2. Scheme of the fumarated Pressure sensitive adhesive (PSA – 1)

2.5 Characterization of Pressure Sensitive Adhesives (PSAs)

2.5.1 Peel strength

The peel strength was measured by coating the adhesive on a thin film polyethylene and then the film was cured in the oven for 1 h at 100°C. The film was then pasted on the glass plate. Common peel tests include the t-peel test, 180° peel test.

2.5.2 Shear test

The shear strength was measured by coating the material on the steel sheet. The time taken for the tape to move a known distance was as the indication of shear strength.

2.6 Characterization of Fiber Reinforced Bio Polyester Composite

2.6.1 Attenuated –transfer infrared spectra – AT-IR

The ATIR spectral analysis of the fiber reinforced Poly (O-PTF)–co-styrene poly ester was done by Perkin Elmer 597 infrared spectrophotometer.

2.6.2 Mechanical properties

2.6.2.1 Tensile test

Tensile strength of the fiber reinforced Polyester composites was determined using Dumbell shaped cut from the specimens, using ISO 527-2:1993 (E) specimen type SA die and tensile test using 100 N load crosshead speed = 100 m/min was done the values represented are an average mean of about 6 - 10 samples.

2.6.2.2 Hardness

Composite hardness (Shore A) was determined using Bareiss durometer.

2.6.3 Thermal studies

2.6.3.1 Thermo gravimetric analysis (TGA) and Differential thermal analysis (DTA)

The thermal properties of the fiber reinforced polyester resin prepared was determined by Differential thermal analysis (DTA) and Thermo gravimetric analysis (TGA) using SDT -2960

(Simultaneous DTA – TGA), TA instruments inc. derived from ASTM E – 1131 -03.

2.6.3.2 DSC characterization

Glass transition temperature of castor oil fiber reinforced polyester resins was determined by DSC based on ASTM U 1356-03 using DSC-2920 Ta instruments Inc, with temperature range – 50 to 130°C, at heating rate 10°C/min

2.6.3.3 Hydrolytic stability test

Hydrolytic stability test of the castor oil fiber reinforced polyester was carried out according to ASTM D 3137.

2.6.3.4 Chemical resistance test

Chemical resistance test for the castor oil fiber reinforced polyester resins was carried out according to ASTM C267.

3. RESULTS AND DISCUSSION

3.1 Spectroscopic Characterization of Fumarated Triglyceride Castor Oil Resin

3.1.1 UV-Vis Spectra

Fig. 3 shows the UV-Vis spectrum of castor oil, hydroxylated resin and O-PTF resin. The UV-Vis spectrum shows band at 265 nm for castor oil which is blue shifted to 243 nm for hydroxylated castor oil resin due to the presence of hydroxyl groups in the castor oil and it is again red shifted to 267 nm for fumarated castor oil resin. The substantial red shift in fumarated castor oil resin indicated the presence of conjugated double bond (K-band) in the fatty acid molecule and confirms the formation of O-PTF resin.

3.1.2 Infrared spectroscopy

Fig. 4 shows the FT-IR spectra of castor oil, hydroxylated oil and fumarated castor oil. In all hydroxylated triglyceride resins, the FT-IR spectra showed a strong absorption band at 3461.99 cm^{-1} , due to the present of free - OH group in the molecule. The corresponding peak is drastically reduced in the O-PTF bio polyester resin indicated the - OH group get substituted. A strong absorbance band in 2925 cm^{-1} is due to the presence of C=O of the glyceryl ester moiety. A strong band in 2850 cm^{-1} is due to the symmetrical -C-H bond in -CH₂- group of the side chain.

3.1.3 NMR Spectra

Fig. 5 shows the NMR spectra of castor oil and fumarated castor oil. The peaks at 4.1 - 4.3 ppm originate from the protons in the methylene groups of the triglyceride. The peak at 5.3ppm indicates the presence of olefinic protons in the oil. This peak disappeared in the hydroxylated resin showing that the olefinic double bond is replaced by the hydroxyl group and reappeared in the fumarated resins due to the presence of unsaturated group. The protons in the - CH₂ - group between the C=C double bonds appear at 2.7 - 2.8 ppm. This peak is shifted to 3 - 3.2 ppm in the hydroxylated resin due to the deshielding caused by the oxygen atom of the hydroxyl group.

3.2 Characterization of Pressure Sensitive Adhesives

3.2.1 AT-IR spectrum of pressure sensitive adhesives

The IR spectrum (Fig. 6) of the surface of the PSA clearly indicates the significant disappearance of peak at $1,645\text{ cm}^{-1}$ and reduction of peak at 981.59 cm^{-1} for the unsaturated double bonds of poly (castor oil fumarate) resin. This is due to the cross linking reaction of O-PTF with vinyl monomers. The strong peaks observed at $1,721\text{ cm}^{-1}$ for the ester groups indicate that the surface of the cured sample is comprised predominantly with ester linkages.

3.2.2 Shear test

Shear test is used to characterize the adhesion properties of tapes for the assessment of shear strength. It gives the ability of a tape joint to resist a load applied in shear mode. It was noted that the curing time increases, shearing time increases to a large extent. Fig. 7 shows the shear test of PSA-1 and PSA-2. From the plot, PSA-2 has higher shear strength compared to PSA-1. It is also noted that the synthesized pressure sensitive adhesives have greater shear strength compared with other reported adhesives [16].

3.2.3 Peel test

Fig. 8 shows the peel test of Pressure sensitive adhesive PSA-1 and PSA-2. It is evaluated by the tensional force required to remove the adhesive tape. It depends on various factors

such as viscoelastic properties of the adhesive, stiffness of the adherent rate of separation, temperature etc. PSA-2 gives high peel strength at 50 peel rate in comparison to all other composition.

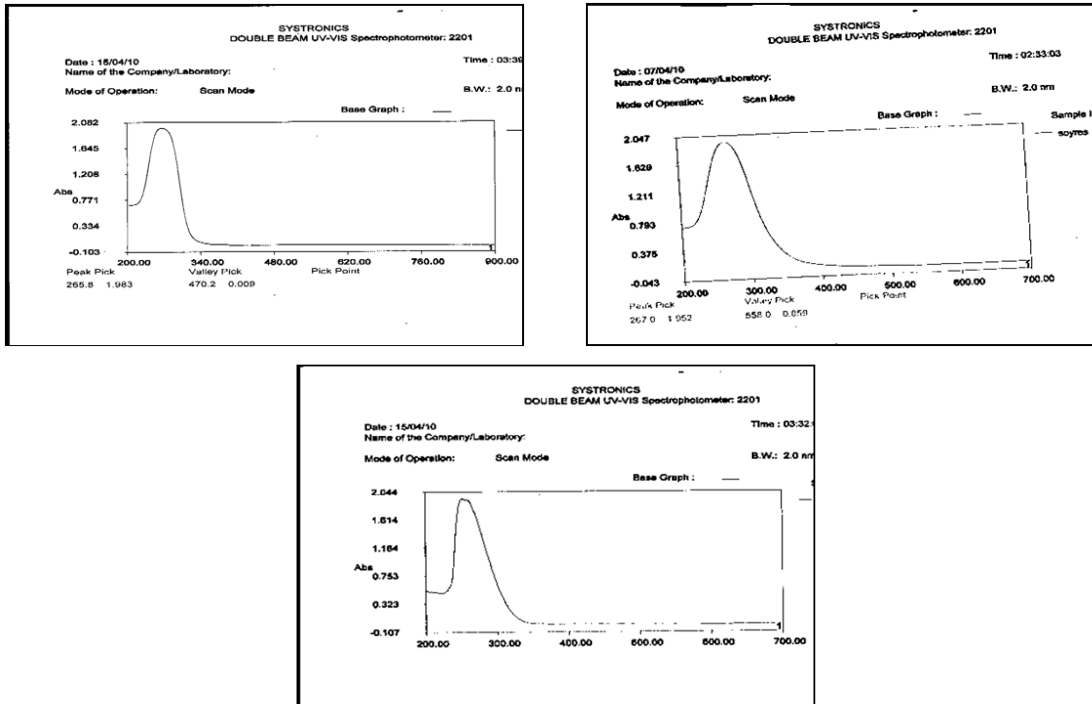


Fig. 3. UV-Vis spectra of castor oil, hydroxylated oil, O-PTF resin

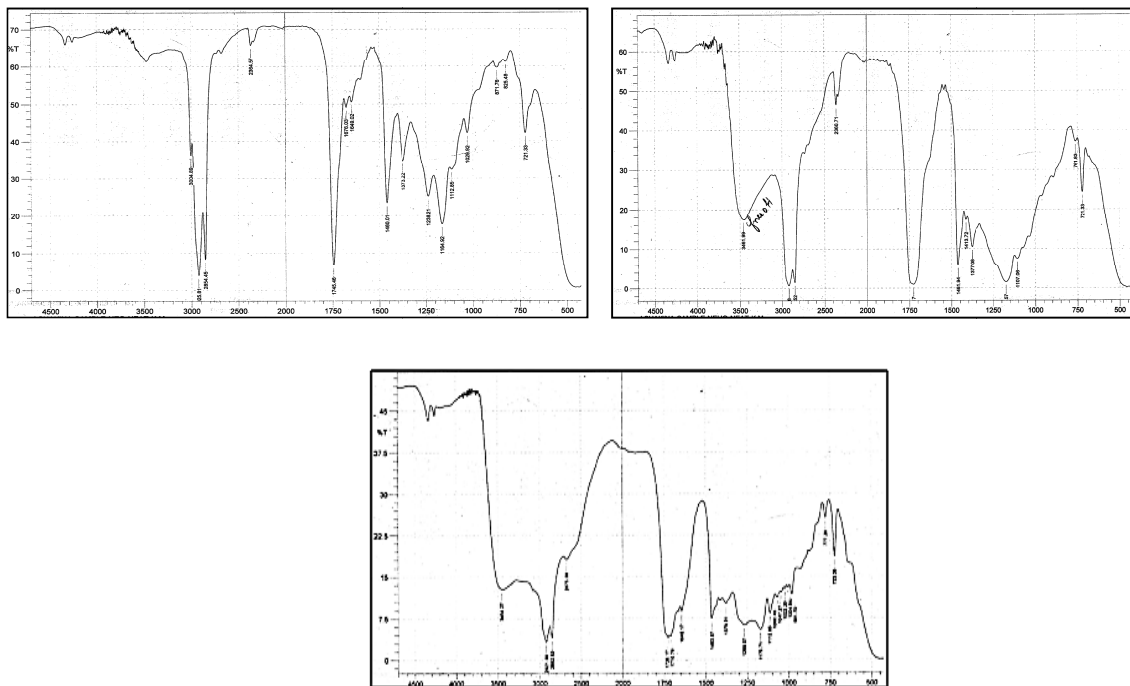


Fig. 4. FT-IR spectra of castor oil, hydroxylated oil, fumarated castor oil

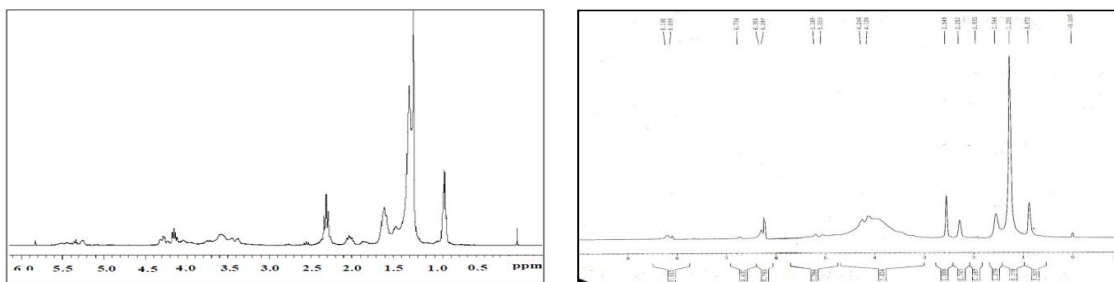


Fig. 5. NMR spectra of castor oil and fumarated castor oil

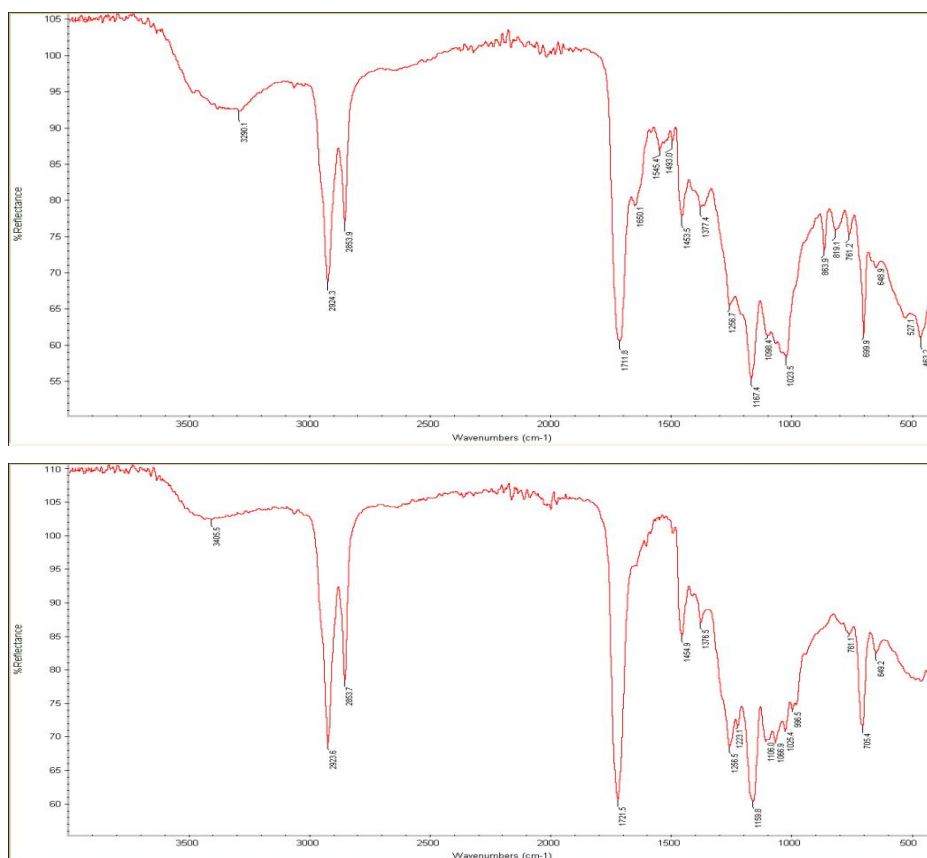


Fig. 6. FT-IR spectra of PSA-1 and PSA-2

3.2.4 Hydrolytic stability

Hydrolysis is one of the dominant mechanisms for the degradation of PSA in the aqueous environment. Hydrolysis can be considered as the reversal of condensation PSAs are susceptible to hydrolytic attack. The main hydrolytic attack is centered on the ester unit in the triglyceride unit. The weight loss of the present PSAs samples aged in water, methanol and sodium chloride solution (1N) are given in Table 1. Aging in sodium chloride solution (1N) does not influence the hydrolytic stability. There

is no weight loss in any of the PSAs in sodium chloride solution. Table 1 shows the Hydrolytic stability of PSA-1 and PSA-2.

3.2.5 Antimicrobial studies

Fig. 9 shows the antimicrobial activity of PSA-1 and PSA-2. Both the test material PSA-1 and PSA-2 exhibited a little antimicrobial activity against gram positive *S. aureus* and gram positive *E. coli* and the zone of inhibition varied from 5 mm-6 mm in size inclusive of the material.

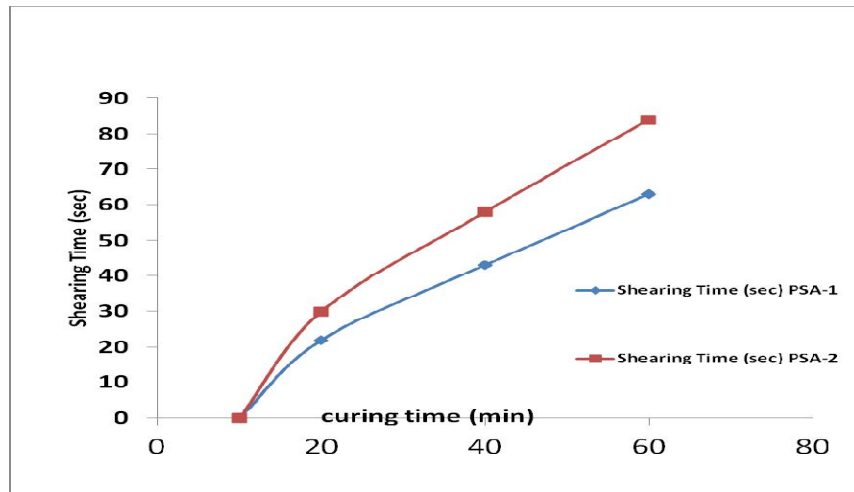


Fig. 7. Shear test of Pressure sensitive adhesive PSA-1 and PSA-2

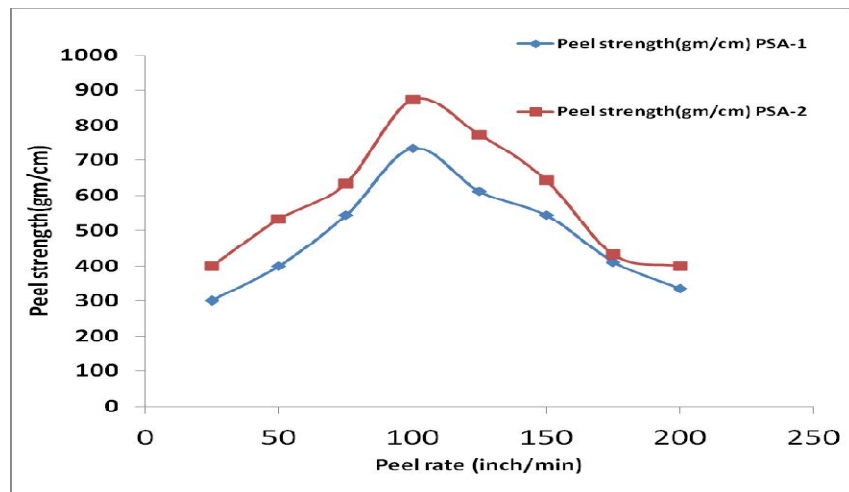


Fig. 8. Peel test of Pressure sensitive adhesives PSA-1 and PSA-2

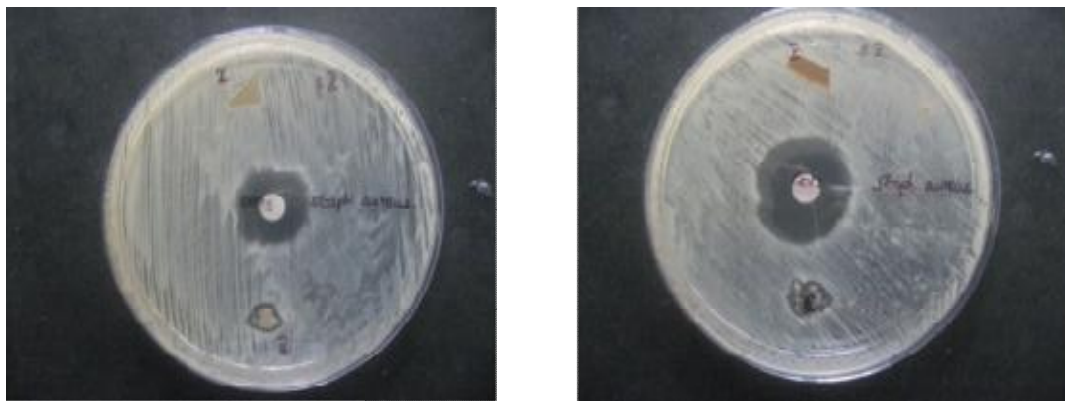


Fig. 9. Antimicrobial activity of PSA-1 AND PSA-2

3.3 Characterization of Fiber Reinforced Polyester Composites (FRPC)

3.3.1 Castor-sisal fiber reinforced polyester (CSFRP)

Fig. 10 shows the AT-IR spectra of new novel elastomeric Castor-sisal fiber reinforced polyester and it showed the following absorption band. The AT-IR spectrum of Fiber reinforced bio polyesters from O-PTF resins of castor oil with styrene monomer responses at 2921.9 cm^{-1} is due to the aliphatic C-H group in the chain, the peak at 1730 cm^{-1} (C=O stretch in esters) and peak at 1454 cm^{-1} is due to $-\text{CH}_2$ linkages and the peak at 1372 cm^{-1} is due to $-\text{CH}_3$ group and 3500 cm^{-1} is due to $-\text{OH}$ group.

3.3.2 Scanning electron microscope

Fig. 11 shows the SEM images of castor oil-sisal fiber reinforced polyester. The morphological results show that there is a proper mixing of fiber with the resin in the bio composites thus synthesized. Morphological studies evidently demonstrate that when polymer resin matrix is reinforced with different loadings of fiber, morphological changes take place depending upon the interfacial interaction between the varying loading of fiber and the resin matrix.

3.4 Mechanical properties

3.4.1 Hardness

The shore - A hardness is defined as the resistance offered by the rubber and similar materials to the penetration of truncated cone (Shore-A). The Shore - A hardness of the soy oil biopolyesters shows the higher hardness among all the biopolyesters prepared from vegetable oil castor oil. Fig. 12 shows the results for polyester/sisal and polyester/jute composite hardness at different compositions (5%, 10%, 20%, 30% and 40%) of fiber. From that, the composite hardness increased with the increased sisal/jute fiber content in the composite. Thus, the sisal and jute fiber is acting as a stiffener for polymer matrix respectively.

3.4.2 Tensile test

The tensile strength of the polyesters is largely influenced by the presence of aromatic groups, ether groups, long alkyl chain, dangling chains, branching and cross linking and also degree of hydrogen bonding. The addition of styrene comonomer to the macro triglyceride monomer introduces some degree of flexibility in the rigid polymer. Fig. 13 shows the tensile strength of castor-sisal fiber reinforced polyester composite at different composition of sisal fiber. Increase in the composition of fiber increases the tensile strength of FRPC.

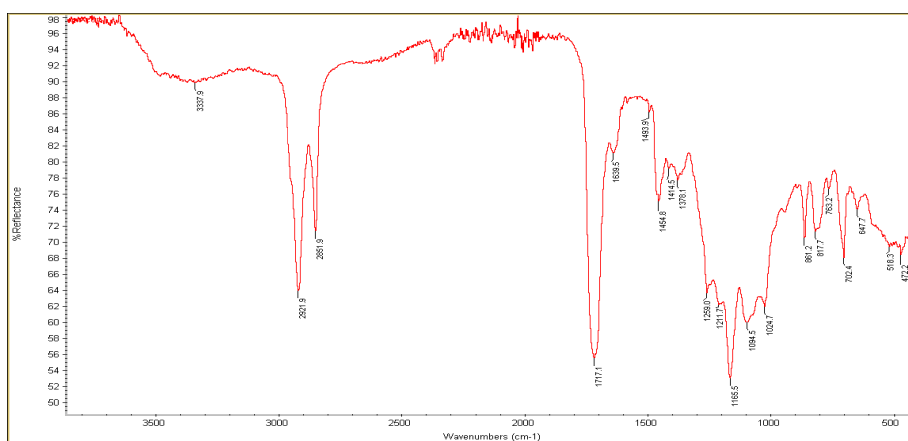


Fig. 10. AT-IR spectra of new novel elastomeric Castor-sisal fiber reinforced polyester

Table. 1 Hydrolytic stability

| Polyesters | Weight loss (%) | | | |
|------------|-----------------|---------|-------------------------------|---------------|
| | Water | Ethanol | Sodium chloride solution (1N) | Boiling water |
| PSA1 | 1.11 | 0.01 | 0.06 | 5.53 |
| PSA2 | 1.04 | 0 | 0.05 | 2.02 |

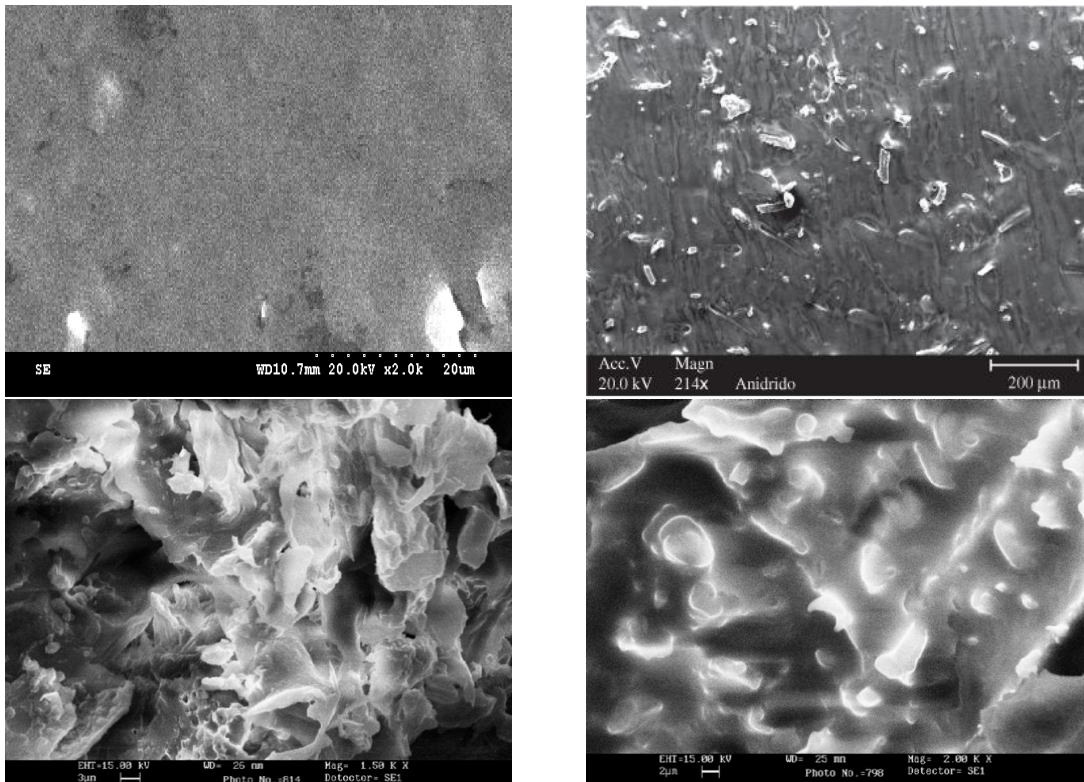


Fig. 11. SEM image of castor oil sisal fiber reinforced polyester

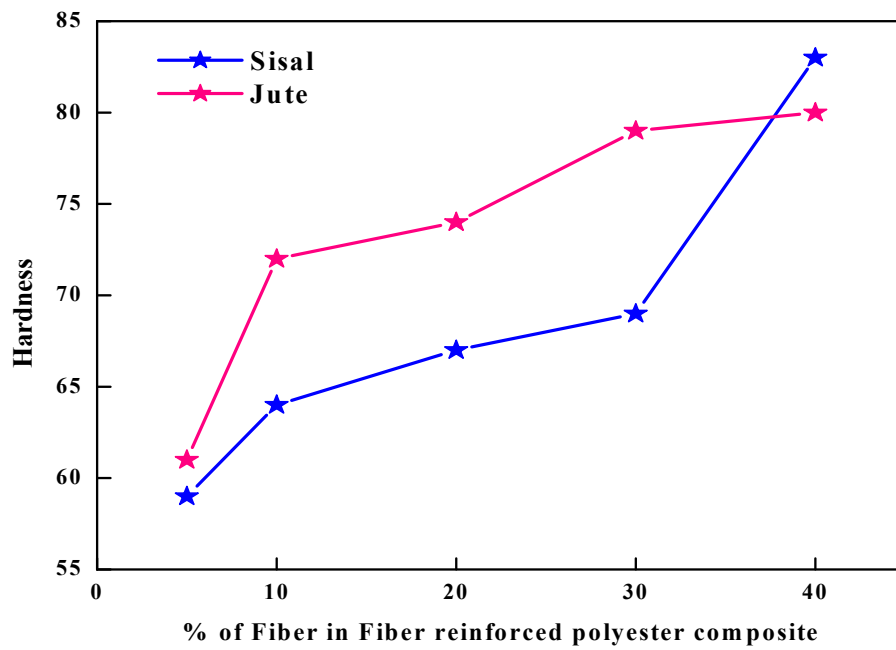


Fig. 12. Hardness of FRPC at different compositions of fiber

3.5 Thermal Studies

3.5.1 Thermo Gravimetric Analysis of sisal fiber reinforced composite

Fig. 14 TGA-DSC curve for SFRPC. Thermal properties of cross linked biopolyesters are influenced mainly by molecular weight between cross links and the degree to which segments form stiff sequences and elastically active branch points. The segments constituting stiff sequences are due to the vinyl monomers cross linked with fumarate groups. Free aliphatic long chains can impart flexibility to the cross linked mass. The appearance of peaks for the softening, decomposition and cross linking was observed for the cross linked biopolyesters. During degradation, the reactions of chain scission and

further cross linking occurred simultaneously with an increase in the number of chemical bonds in the network that led to the char structure formation at the end of the process. Table 2 shows the TGA of castor sisal fiber reinforced polyester (CSFRP).

3.5.2 DSC Analysis of Castor sisal fiber reinforced polyester (CSFRP)

Fig. 15 shows the DSC curve for Castor sisal fiber reinforced polyester (CSFRP). Differential scanning calorimetry (DSC) provides a rapid method for determining polymer crystallinity based on the heat required to melt the polymer. From the Tg value it is semi crystalline in nature. The glass transition temperature of CSFRPC is 122°C and the melting enthalpy is 564.22°C.

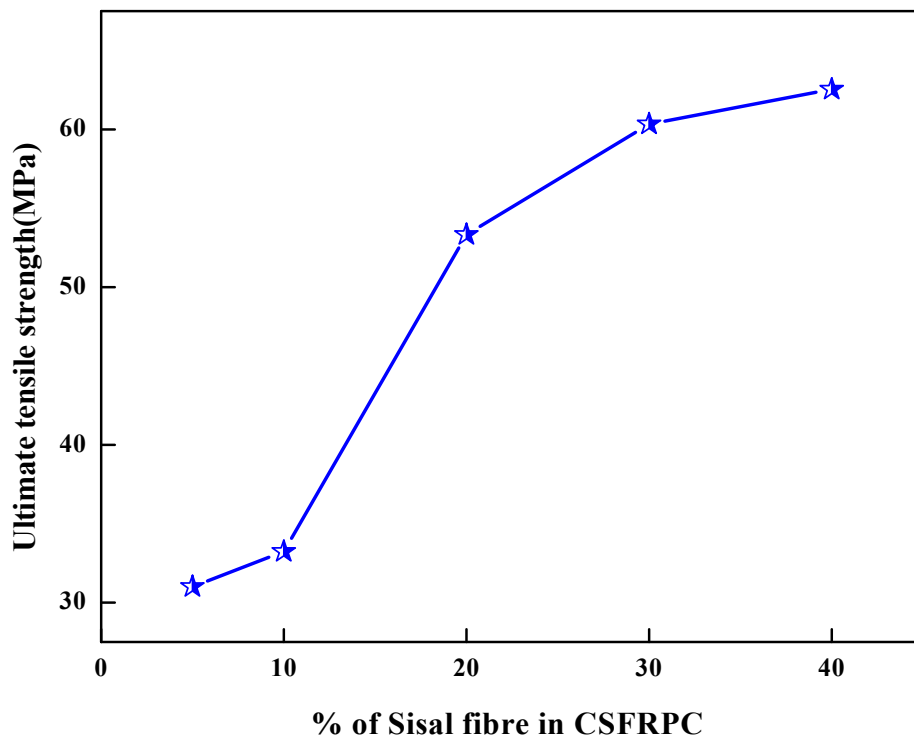


Fig. 13. Tensile strength of CSFRPC at different fiber compositions

Table. 2 TGA of castor sisal fiber reinforced polyester (CSFRP)

| S. no. | Sample code | IDT°C | % wt loss | FDT°C | % wt loss | Final residue |
|--------|-------------|----------|-----------|-------|-----------|---------------|
| 1 | SFRPC | 219.72°C | 9.63 | 593°C | 98.408 | 3.005% |

3.6 Chemical Properties of Castor-fiber Reinforced Composite Polyester

3.6.1 Chemical resistance test

The weight loss of polymers was determined in hostile acidic, basic and oxidation medium. The

degradation of Polyester resins in dilute hydrochloric acid (1N), Sodium hydroxide and 30% hydrogen peroxide were used. It was found that the CSFPC and CJRPC are highly stable against all reagents. Table. 3 shows the chemical resistance test of CSFPC and CJRPC.

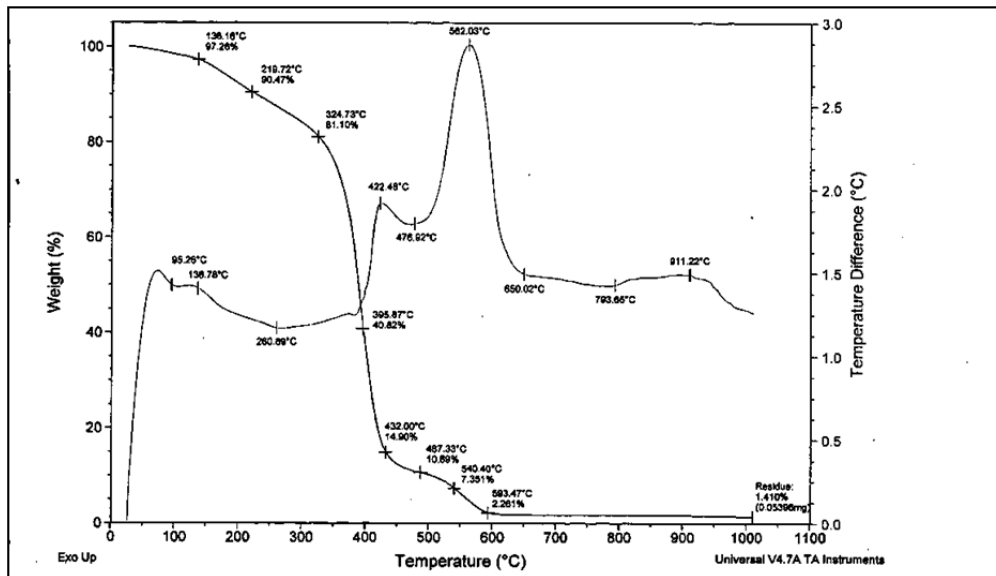


Fig. 14. TGA-DSC curve for SFRPC

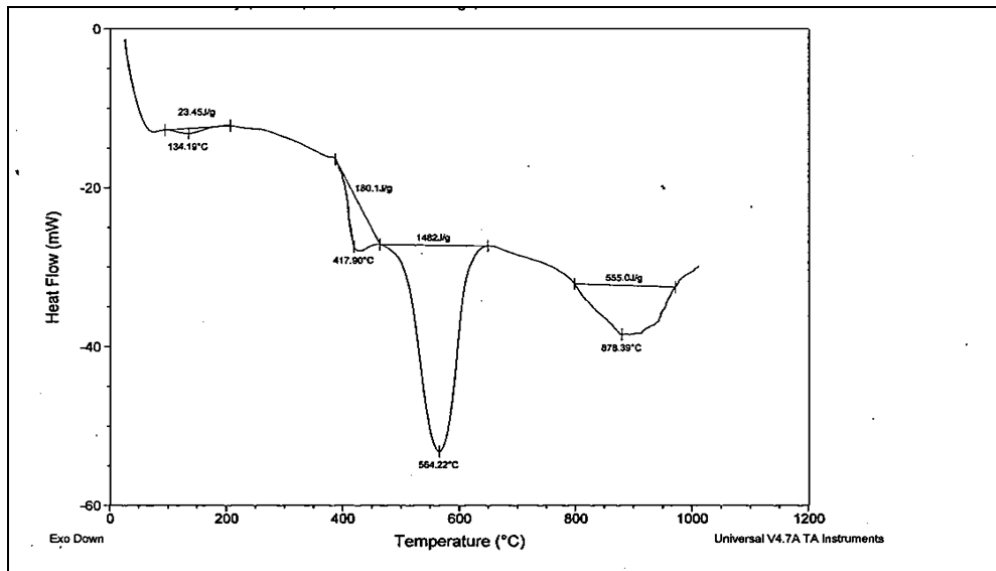


Fig. 15. DSC curve for Castor sisal fiber reinforced polyester (CSFRP)

Table 3. Chemical resistance test

| S. No. | Samples | Weight loss (%) | | | | |
|--------|---------|-----------------|------------|-------------|-----------------------------------|------------------|
| | | HCl | 0.1 N NaOH | 30% alcohol | 30% H ₂ O ₂ | H ₂ O |
| 1. | CSFPC | 1 | 12 | 1.4 | 0 | 0 |
| 2. | CJRPC | 1 | 13 | 1.47 | 0 | 0 |

4. CONCLUSION

Copolymerization of O-PTF resin with VA and MA without any solvent leads to a cross linked polymer with high molecular weight and good resistance to chemicals. Degradation studies show that the pressure sensitive adhesives PSA-1 and PSA-2 are biodegradable. Both of them may be suitable for non-occlusive PSA tapes there by decrease the level of skin irritation considerably and also minimize the incidence of bacterial infection. It also serves as a bacteriostatic to prevent the growth of bacteria on the plastic surface. Sisal and Jute fibers are used for the synthesis of fiber reinforced polyester composites. The results suggest that these composites can be future materials for the fabrication of eco-friendly materials.

5. FUTURE SCOPE

This study leaves wide scope for future investigations. It can be extended to newer composites using other reinforcing phases and the resulting experimental findings can be similarly analyzed. The evaluation of sisal fiber reinforced polyester resin composite has been a much less studied area. There is a very wide scope for future scholars to explore this area of research.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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