INTERNATIONAL JOURNAL OF PURE AND APPLIED RESEARCH IN ENGINEERING AND TECHNOLOGY

A PATH FOR HORIZING YOUR INNOVATIVE WORK

SPECIAL ISSUE FOR INTERNATIONAL LEVEL CONFERENCE "ADVANCES IN SCIENCE, TECHNOLOGY & MANAGEMENT" (IC-ASTM)

SYNTHESIS AND THERMO-MECHANICAL CHARACTERIZATION OF PANI-AC COMPOSITES SUPPORTING WITH POLYESTER MATRIX

V. T. BHUGUL.

Department of Applied Chemistry & Polymer chemistry, College of Engineering & Technology Akola, Maharashtra (India)

Abstract: Variety of blends and composites of conducting polymers such as polyaniline, polypyrrole, polythiophene etc. have been studied for their application in energy storage systems, opto-electronic devices, light emitting diode, nonlinear optical devices, gas and moisture sensor, microwave absorbing materials etc. In present work the composite of polyaniline and finely divided activated charcoal with supporting matrix of unsaturated polyester were synthesized by in-situ polymerization technique .The synthesized composite with different composition were studied for their thermal stability using characterization technics like TGA and DSC. Mechanical properties such as tensile strength, young modulus, % elongation etc were studied with the help of universal testing machine.

Corresponding Author: V. T. BHUGUL

Co Author: -

Access Online On:

www.ijpret.com

How to Cite This Article:

V. T. Bhugul, IJPRET, 2017; Volume 6 (2): 371-377

PAPER-QR CODE

INTRODUCTION

Among conducting polymers Polyanilene (PANI) an inherently conducting polymer and its copolymers have been widely used to study due to ease of their protonic acid doping in the emeraldine forms and environmental stability in both doped and undoped forms. PANI has attracted tremendous attention because of its technological application potential in several areas.[1-4] There are two main groups of applications for these polymers. The first group utilizes their conductivity as its main property this includes antistatic coating, conductive adhesives, electromagnetic shielding, printed circuit boards, artificial nerves and aircraft structure etc. The second group utilizes their electro activity it includes molecular electronics, electrical displays, sensors, rechargeable batteries, electromechanical actuators and smart structures [5-8]. The extended p systems of conjugated polymer are highly susceptible to chemical or electrochemical oxidation or reduction. These alter the electrical and optical properties of the polymer, and by controlling this oxidation and reduction, it is possible to precisely control these properties. Since these reactions are often reversible, it is possible to systematically control the electrical and optical properties with a great deal of precision. It is even possible to switch from a conducting state to an insulating state. PANI is typically phenylene based polymer having a chemically flexible –NH- group in the chain flanked either side by phenylene ring. The protonation, deprotonation and various physico - chemical properties of polyaniline are due to presence of this group. Polyaniline (PANI) is a unique and promising candidate for potential applications because of its stability in air and solubility in some organic solvents (Angelopoulos *et al* 1988; Cao *et al* 1992). It also exhibits dramatic changes in its electronic structure and physical properties on protonation (Mexiang Wan *et al* 1991, 1992). Polyaniline is the oxidative polymeric product of aniline under acidic conditions and has been known since 1862 as aniline black. The interest in this conducting polymer stems from the fact that many different ring- and nitrogen-substituted derivatives can be readily synthesized and that each of the derivatives can exist in several different oxidation states which can in principle be "doped" by a variety of different dopants either by non-redox processes or by partial chemical or electrochemical oxidation. These properties, combined with the fairly high conductivity after doping, their ease of synthesis and processing, satisfactory environmental stability, simple doping by protonic acid, its polymerization proceeds with high speed and the resulting salt is quit stable and show relatively high level of conductivity. It is the only known conducting polymer with nitrogen atoms occupying the bridging to the benzenoid and quinoid ring in its backbone structure and playing an important role to the π – bond formation and the electrical inter chain conduction. The relatively low cost of the starting materials strongly suggest their significant potential technological applicability.

The processibility of PANI has been achieved by making conductive composites or blends of PANI with conventional polymer like polystyrene (PS), polyesters (UPS) polyvinyl acetate (PVAc), polyvinyl chloride (PVC) wherein the composites retain the thermo-mechanical properties of the conventional polymer and conducting polymer.[9,10]. Polyester matrices have been in use for the longest period in the widest range structures because of easy handling, balanced mechanical and chemical characteristics and cheap price. Polyesters cure with the addition of a catalyst such as a peroxide resulting in an exothermic reaction, which can be initiated at room temperature.

In this chapter, unsaturated polyester resin was preferred due to their good range of thermo-mechanical properties, low cost, corrosion resistance and low molecular weight. Therefore, polyesters were suitable for variety of applications and are adaptable to the fabrication of large structures. [11-14].

2. Experimental

2.1 Materials

The aniline monomer, Ammonium peroxodisulfate and dimethyl sulfoxide (DMSO) purchased from Merck. India. Unsaturated polyester resin from Wilson k. laboratories, Mumbai were used as received. HCl, Methyl ethyl ketone peroxide (MEKP). Cobalt octet was used as a hardener purchased from S.D. fine chemicals. India. All chemicals were of analytical grade and used as received.

2.2 Synthesis of polyaniline Emeraldine Salt

For the synthesis of polyaniline (ES) , Ammonium peroxodisulfate $(NH_4)_2S_2O_8$ (12.3 g, 0.054 mol), was dissolved in 70 ml of 1M HCl in a 500 ml beaker and kept at 5^0C using ice bath. Aniline was purified by double distillation at reduced pressure (5 g, 0.054 mol), was dissolved in 75 ml of 1M HCl in another 500 ml beaker and also kept at 5^0C. Then, ammonium peroxodisulfate solution was added slowly to the aniline solution over a period of 1 minute. The flask was left for 30 minutes, during which the temperature remained constant at 5^0C. The solution started to take on a blue-green tint after one to three minutes, then became intense blue green with a coppery glint. After 30 minutes, the precipitate was collected on a Buchner funnel and flask using a water aspirator. The precipitate cake was washed portion wise (80 ml/portion) with deionized water until the filtrate became colorless. The liquid level in the filter should be constantly adjusted to be above the top of the precipitate. This prevented cracking of the precipitate cake, and hence avoid inefficient washing of the precipitate. In order to remove oligomers and other non-polymeric impurities the precipitate was washed thoroughly with methanol and deionized water. After washing, the precipitate remained under section for near about 10 minutes until significant cracking of the moist filter cake occurred. This cake was dried at 50-60^{0}C in an oven, powder in mortar and stored for processing, the yield was found about 92%.

2.3 Synthesis of Polyaniline-Activated charcoal composite with supporting matrix of polyester resin

The activated charcoal particles were pretreated by boiling in 4 mol/dm^{-3} HNO$_3$ solution for 4 hours, then washed with deionized water until pH~ 7. The 0.1, 0.2, 0.3 and 0.4 g. amount of treated Activated charcoal (AC) was dispersed in   1 mol/dm^{-3} H$_2$SO$_4$ solutions by ultrasonic over 1 h, then the solution was transferred to magnetic stirring apparatus equipped with an ice bath. Aniline monomer was then added to the above suspension of carbon particles. 20 mL of 1 mol/dm^{-3} H$_2$SO$_4$ solution containing APS (molar ratio ANI/ APS 1: 1.2) was slowly added to the suspension keeping constant magnetic stirring, at a reaction temperature of 0–5 ^{0}C for 15 min. After further 6 hours stirring, the resulting green suspension was filtered and rinsed several times with distilled water and methanol until filter became neutral and colorless. The obtained powder was dried in a vacuum oven at 60 ^{0}C for 24 hours.

The resulting composite were added in a 100 ml of unsaturated polyester resin and thoroughly mixed half an hour to get homogeneous mixture and then methyl ethyl ketone peroxide (MEKP) was used as a hardener, as an amount of 1.5 wt. % of USPR was added, the mixture was then poured into the mould and kept it 5-6 hours for drying. After drying the composite were released from the mould in sheet form. Samples were prepared by addition of varying quantity of PANI-AC composite in USPR.

3. Result and Discussion
3.1 Thermo gravimetric analysis

The figure 3.1(a) and 3.1(b) shows the TG diagram of PANI-USPR composite and composite samples having different loading of AC filler. The TG thermogram shows that, the initial mass loss between 50 to 130 ^{0}C is due mainly to release of moisture and acid dopant from the surface of PANI. Further mass loss taking place at a slower rate up to 300 ^{0}C, primarily released of dopant. The sharp mass loss beginning for nearly 370 ^{0}C. For blend the initial release of moisture and dopant take place at relatively same rate as that of pure PANI up to 120 ^{0}C. Slower rate in weight loss above 120 ^{0}C is believed to be due to further release of dopant and continuing till beyond 600 ^{0}C presumably corresponding to large scale thermal degradation. The same trend is observed in other blend samples. This trend of weight loss matches with the thermal degradation of PANI.
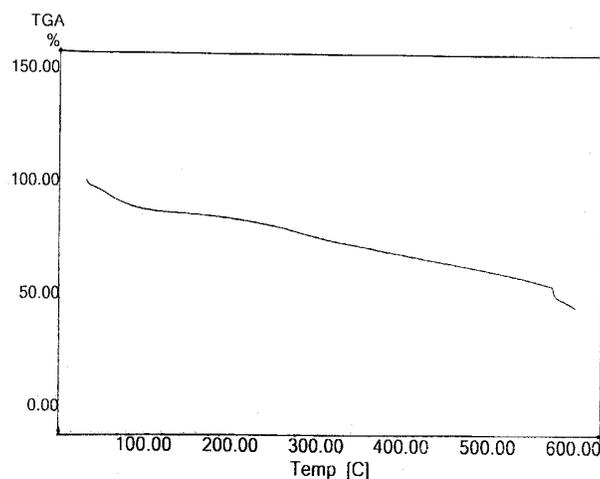

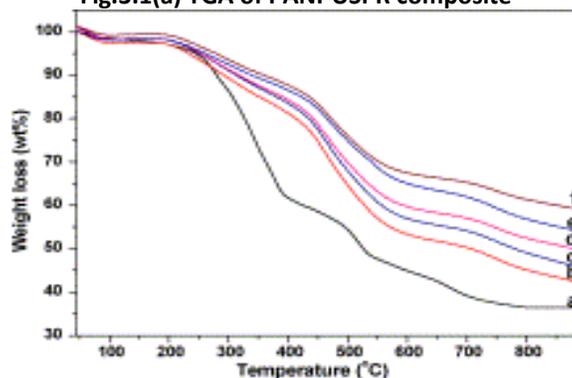**Fig.3.1(a) TGA of PANI-USPR composite**

Fig.3.1 (b) TGA diag. of PANI-USPR-AC composites

3.2 Mechanical Properties

For measurement of mechanical properties such as tensile strength, tensile stress, % elongation and Young's modulus specimen samples of blends with activated charcoal filler loading are prepared. Initially the moisture present in activated charcoal was removed by keeping it in hot air oven at 70 ^{0}C temperature for 1 hour .The in-situ blend of PANI-USPR blend was prepared in non-aqueous medium and in this blend different wt % of activated charcoal fillers were added and curing of blend takes place by addition of MEKP and cobalt octatate as initiator and

374

accelerator respectively. The test specimens in the form of thin sheet of width of narrow section 4.4 mm, distance between grips 50 mm, total length of specimen 110 mm, thickness of specimen 2.05 mm. The tensile strength at break point calculated by dividing the maximum load in Newton's by thr original minimum cross sectional area of the specimen in square meters. % Elongation was calculated by using following formula;

$$\text{Elongation at break} = \frac{(\text{Change in the gauge length at break/ yield}) \times 100}{\text{original gauge length}}$$

Mechanical properties are studied by use of universal testing machine (ASTMD 882 method). Table 1.1 shows tensile properties of blend with AC fillers and figure 3.2(a), 3.2(b) and 3.2(c) shows effect of fillers on tensile strength, % elongation and young's modulus respectively.

Table-1.1 Tensile properties of specimens

Sample	Wt % of AC	Tensile Strength at peak load	% Elongation at break load	Yield Stress	Young's Modulus
PANI-AC-1	0	453.28	6.70	323.07	5220.94
PANI-AC-2	10	614.77	8.40	612.14	6285.13
PANI-AC- 3	20	515.60	6.70	513.40	8285.13
PANI-AC- 4	30	480.32	6.40	431.42	9841.68
PANI-AC- 5	40	517.96	6.00	514.94	9021.40

Unit- Kg/cm^2

The figure 3.2(a) shows the behavior of tensile strength of in-situ blend using AC fillers, in this case there is decrease in tensile strength is observed. This decrease in tensile strength may be attributed to change in effective cross sectional area brought about by AC as filler in dispersed phase.
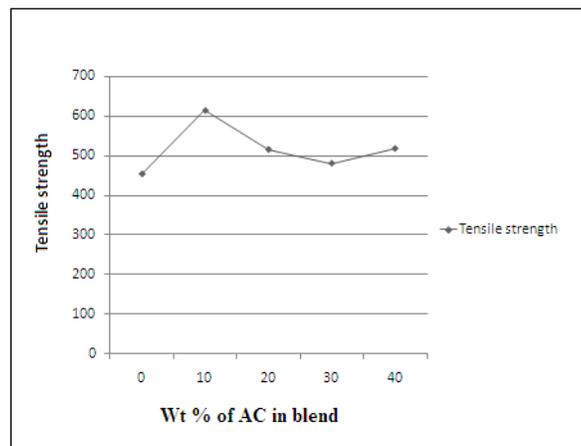

Figure 3.2(a) Effect of AC filler on Tensile strength of Composite

Figure 3.2(b) shows the % elongation of blend, there is marginal decrease in % elongation is observed. Figure 3.2(c) shows young's modulus of in-situ blend in which there is increase was observed up to 30 at % of AC and decreased on increasing wt % of AC.
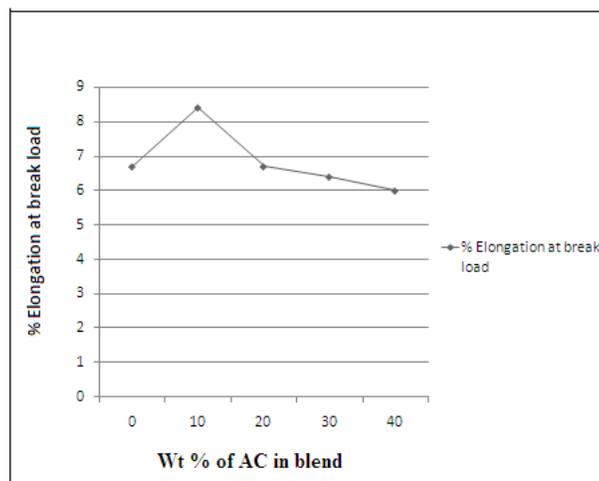
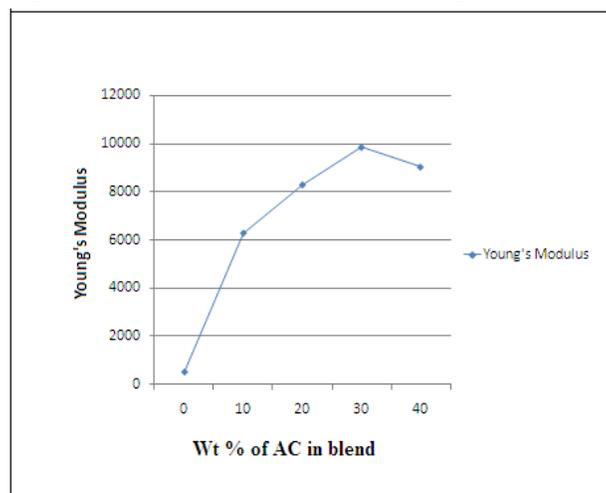**Figure 3.2(b) effect of AC filler on % Elongation of blend**

Figure 3.2(c) Effect of wt. % of AC in blend on young's Modulus

This decrease in modulus values at higher loading indicates that there may be reduction in stiffness of the blend sample. At this loading AC particles may acts as lubricating agent facilitating ductility in the composite/ blend at higher loading.

4. References

1. C. Barbero, H.J. Salavagione, D. F. Acevedo, D.E. Grumelli, F. Garay, and M.C. Miras, Electrochimica Acta, 49,
1. 3671 (2004).
2. E. Erdem, M. Karakitsla and M. Sack, European Polymer Jn., 40, 785 (2004).
3. H. V. Rasika Dias, R. M. Gamini Rajapakse, D. M. Milan Krishantha, X.Wang, M. Fianchini and R. L. Elsenbaumer, Journal Materials Chemistry, 17, 1762 (2007).
4. E. Erdem, M. Sacak and M. Karakisla Polymer International 39 153 (1996).
5. P.F.W. Simon, R. Ulrich, U. Wiesner, Chem. Master 13, 3464 (2001)
6. M.R. Sabotakin, A.M. Maharramov, M.A. Ramazanov, Nature and Science 5(3), 67(2007).

7. M.R. Sabotakin, A.M. Maharramov, M.A. Ramazanov, Jn. of American Science, 3(4), 40(2007).

8. M.R. Sabotakin, A.M. Maharramov, M.A. Ramazanov, Jn. of American Science, 3(4), 30(2007).

9. A.K. Bledzki and J. Gassan, J. Prog Polym. Sci. 24, 221-274, (1999).

10. F. W. J. Van Hattum, F. Regel, and M. Labordus, Plast Rubber Comp., 40, (2011) 93-99.

11. F. L. Mathews and R. D. Rawaling. Composite materials, Engg. And Sci. Chapman and Hall, (1994).

12. M. Shwartz. Handbook of structural ceramics. (1992).

13. D. Hull and T.W. Clyne. Jn. composite Mat. 35, (1999) 99-110.

14. M. Wan and J. Yang, J. Appl. Polym Sci., 55, (1995) 399.