Industrial Crops and Products 58 (2014) 251–258

Contents lists available at ScienceDirect

Industrial Crops and Products

journal homepage: www.elsevier.com/locate/indcrop

Microwave assisted synthesis of polyacrylamide grafted soya peptone and its application as water soluble adhesive

D.Mahto[a], Priti Rani[b], Sumit Mishra[b], Gautam Sen[b,*]

[a] Department of Mechanical Engineering, Birla Institute of Technology, Mesra, Ranchi 835215, Jharkhand, India
[b] Department of Applied Chemistry, Birla Institute of Technology, Mesra, Ranchi 835215, Jharkhand, India

ARTICLE INFO

Article history:
Received 1 December 2013
Received in revised form 30 March 2014
Accepted 10 April 2014

Keywords:
Soya peptone
Adhesive
Microwave assisted synthesis
Graft co polymer

ABSTRACT

Synthesis of polyacrylamide (PAM) grafted soya peptone was performed by *the microwave assisted method* using ceric ammonium nitrate (CAN) as free radical initiator. The synthesis was optimized by varying the acrylamide and CAN concentration. The grafting of the PAM chains on the peptone backbone was confirmed through various physicochemical techniques-intrinsic viscosity study, FTIR spectroscopy, elemental analysis (C, H, N, S and O), SEM and TGA. The applicability of this synthesized graft copolymer as a water soluble adhesive has been studied by fabricating a *single lap joint* of two wooden blocks using an aqueous paste of the graft copolymer as adhesive. After curing, the breaking point of the joint was determined using an UTM.

1. Introduction

Proteins are the basic building blocks of life. Proteins are long polymeric chains of α amino acids. Peptones are prepared by the enzymatic or acidic hydrolysis of proteinaceous material. These hydrolysates contain secondary protein derivatives such as polypeptides, dipeptides and amino acids. They provide a readily assimilable source of nitrogen which is water soluble and does not coagulate on heating. Therefore it is particularly suitable for inclusion in microbiological culture media (Oxoid Manual, 1988. United Kingdom; Brewster and Mcewen, 1958). Hydrolysis of proteins yield meta proteins, proteoses, peptones, polypeptides, and finally the amino acids (Difco Manual, 1964). There are about three types of peptones – meat peptones (such as peptone bacteriological, tryptose, etc.), vegetable peptone (soya peptone), casein and other milk derived peptones (Difco Manual, 1964; Bridson, 1995). Hydrolysis of proteins – breaking them down to their constituent amino acids and peptides, can be achieved by the use of strong acids, strong bases or proteolytic enzymes. This hydrolysis of a protein is a gradual process by which the gigantic molecule of protein is converted into products of successively lower molecular weight.

Soya peptone is a vegetable peptone (Oxoid Manual, 1988. United Kingdom). It is obtained by the papain hydrolysis (enzymatic hydrolysate) of soya flour (Oxoid Manual, 1988. United Kingdom; Difco Manual, 1964; Bridson, 1995) under controlled conditions. Soya flour which is a combination of soy protein and its associated carbohydrate, is available in excess amounts in the United States as a by-product of the soybean industry. It generally contains approximately 50% protein, and the balance is carbohydrates and fats (Thakur et al., 2014). It is an excellent source of vitamins and carbohydrates.

Peptones find extensive use in microbiological culture media to support the nutritional requirements of microorganisms, the production of human and veterinary vaccines, in nutrient media for growing bacteria and fungi, for detection of microorganisms, in fermentation and cell culture industries.

Composite materials are materials made from two or more constituent materials with significantly different physical or chemical properties, that when combined, produce a material with characteristics different from the individual components. The use of bio based materials from a number of renewable resources as a component in composites can provide positive environmental benefits that are highly desired for the green environment. Among the most important advantages of bio based reinforcing materials over traditional materials such as synthetic fibre (glass fibre, carbon fibre) are acceptable specific strength properties, easy availability, low cost, eco-friendliness, lightweight, ease of separation, non-corrosive nature, low density, reduced tool wear, reduced dermal and respiratory irritation, less abrasion to processing equipment, and renewability (Thakur et al., 2013d).

Graft copolymers by definition, consist of a long sequence of one polymer (backbone polymer) with one or more branches (grafts) of

---

* Corresponding author. Tel.: +91 9470137364.
E-mail addresses: gsen06@gmail.com, gsen9@hotmail.com (G. Sen).

http://dx.doi.org/10.1016/j.indcrop.2014.04.013
0926-6690/

another (chemically different) polymer (Odian, 2002; Gowariker et al., 1986). The process of graft copolymer synthesis starts with a preformed polymer (maybe a polysaccharide or a protein or a peptone as in this case). An external agent is used to create free radical sites on this preformed polymer. The agent should be effective enough to create the required free radical sites, at the same time should not be too drastic to rupture the structural integrity of the preformed polymer chain (Sen and Pal, 2009a).

Once the free radical sites are formed on the polymer backbone (i.e. preformed polymer), the monomer (i.e. vinyl or acrylic compound) can get added up through the chain propagation step, leading to the formation of grafted chains. The various methods of graft copolymer synthesis actually differ in the ways of generation of the free radical sites on this preformed polymer.

The most contemporary technique in graft copolymer synthesis involves the use of microwave radiations to initiate the grafting reactions. The superiority of this technique over others has been well discussed in earlier studies (Sen and Pal, 2009a,b; Sen et al., 2010a,b; Mishra et al., 2011a,b, 2012; Mishra and Sen, 2011; Sen et al., 2011; Pal et al., 2011; Thakur et al., 2012, 2013a,b, 2014a,b; Thakur and Singha, 2011).

Microwave based graft copolymer synthesis is further classified into two types.

Microwave initiated synthesis (using microwave radiation alone to initiate grafting) and microwave assisted synthesis (using a synergism of microwave radiation and chemical free radical initiator to initiate grafting). The properties of the grafted product produced can be finely tuned in terms of *percentage grafting*. Thus, the process of grafting can be regarded as *molecular programming* which involves programming of desired properties at the molecular level. Evidently, being tailor made materials, graft co-polymers have a diverse range of applications – from controlled and targeted drug release (Sen and Pal, 2009b; Sen et al., 2010a,b) to algal biomass harvesting (Banerjee et al., 2012) to wastewater treatment (Sen and Pal, 2009a; Rani et al., 2012) to improve the swelling and thermal properties of the biofibres (Thakur et al., 2013c) using free radical-induced graft copolymerization. In this paper we discussed the study of the synthesized novel graft copolymer (SOYP-g-PAM) as adhesive.

1.1. Plan of investigation

The study described in this paper involve *microwave assisted* synthesis of graft chains of polyacrylamide (PAM) onto the backbone of soya peptone, thus resulting in the formation of polyacrylamide grafted soya peptone (SOYP-g-PAM). The synthesis has been carried out using ceric ammonium nitrate (CAN) as the free radical initiator. The synthesis was optimized in terms of percentage grafting by varying the acrylamide and CAN concentration. The applicability of this synthesized graft copolymer as adhesive has been studied by fabricating a *single lap joint* by sticking two wooden blocks using an aqueous paste of the graft copolymer as adhesive. After curing, the breaking point of the joint was determined using a UTM.

2. Materials and methods

2.1. Materials

Soya peptone was supplied by CDH, New Delhi, India. Acrylamide was procured from E.Merck, Germany. Ceric ammonium nitrate was supplied by E.Merck (India), Mumbai, India. Analar grade of acetone and hydroquinone was purchased from S.D. Fine Chemicals, Mumbai, India. All the chemicals were used as received; without further purification.

2.2. Synthesis of graft copolymer

2.2.1. Microwave assisted synthesis of polyacrylamide grafted Soya peptone (SOYP-g-PAM), using ceric ammonium nitrate (CAN) as free radical initiator

1 g of soya peptone was dissolved in 40 ml distilled water. Desired amount of acrylamide was dissolved in 10 ml distilled water and added to the soya peptone solution under mid-high speed stirring, followed by addition of catalytic amount of ceric ammonium nitrate (CAN). Once mixed homogeneously, the reaction mixture was transferred to the microwave reactor (Catalyst^{TM} system CATA 4 R). In the reactor, stirring was continued and irradiated with microwave radiation (700 W) until gelling set in, keeping the irradiation cut-off temperature at 70 °C. Once the microwave irradiation procedure got complete, the gel like mass left in the reaction vessel was cooled and poured in excess of acetone. The precipitated grafted polymer was collected and was dried in hot air oven. Subsequently, it was pulverized and sieved. The synthesis details of various grades of the graft copolymer have been shown in Table 1. The percentage grafting of this microwave assisted synthesized SOYP-g-PAM was evaluated as:

$$\%G = \frac{\text{Wt. of graft copolymer} - \text{Wt. of peptone}}{\text{Wt. of peptone}} \times 100$$

The proposed mechanism of microwave assisted grafting has been depicted in Scheme 1 and the synthesis details of various grades of the graft copolymer have been shown in Table 1.

2.2.2. Purification of the graft copolymer by solvent extraction method

Any occluded polyacrylamide (PAM) formed by competing homopolymer formation reaction was removed from the graft copolymer synthesized as above, by solvent extraction with acetone for 24 h (Sinha and Mishra, 2013).

2.3. Characterization

2.3.1. Intrinsic viscosity measurement

Viscosity measurements of the polymer solutions were carried out with an Ubbelodhe viscometer (Constant: 0.003899) at 25 °C. The viscosities were measured in aqueous solutions. The pH of the solution was neutral. The time of flow for solutions was measured at four different concentrations (0.1%, 0.05%, 0.025% and 0.0125%). From the time of flow of polymer solutions (t) and that of the solvent (t_0, for distilled water), relative viscosity ($\eta_{rel} = t/t_0$) was obtained. Specific viscosity (η_{sp}), reduced viscosity (η_{red}) and inherent viscosity (η_{inh}) was mathematically calculated as:

$$\eta_{sp} = \eta_{rel} - 1$$

$$\eta_{red} = \frac{\eta_{sp}}{C}$$

$$\eta_{inh} = \frac{\ln \eta_{rel}}{C}$$

where ‘C’ represents polymer concentration in g/dl.

Subsequently, the reduced viscosity (η_{red}) and the inherent viscosity (η_{inh}) were plotted against concentration. The intrinsic viscosity was obtained from the point of intersection after extrapolation of two plots (i.e. η_{red} versus C and $\ln \eta_{inh}$ versus C) to zero concentration (Collins et al., 1973). The relation between intrinsic viscosity and percentage grafting has been graphically depicted in Fig. 1.