

ORIGINAL ARTICLE

A novel initiating system for wool grafting

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KEYWORDS

Wool; Redox; Grafting; Acrylic monomers; Characterization **Abstract** This paper describes a new method for the grafting of methacrylic acid (MAA) and other acrylic monomers onto wool fabric in aqueous medium. The novelty principally concerns the chemical approach of the redox grafting reaction that was carried out in the presence of sodium perborate (SPB) initiator. Before the grafting reaction was started, the wool fabric was treated first with a freshly prepared ferrous ammonium sulfate (FAS) solution. The so-treated fabric formed, with SPB, an efficacious redox system capable of initiating grafting of methacrylic acid (MAA) and other acrylic monomers onto the wool fabric. The effect of the polymerization conditions on the polymer criteria, namely, graft yield (%GY), homopolymer (%HP), total conversion (%TC), and grafting efficiency (%GE), was studied. These polymer criteria were found to depend spaciously upon concentrations of the Fe²⁺ ion (activator), SPB (initiator), and MAA, pH of the polymerization medium, duration and temperature of polymerization. The graft copolymerization reaction has also been carried out in the presence of polymerization activators (e.g. reductant transition metal ions) and an inhibitor (e.g. hydroquinone). A suitable mechanism for the grafting processes has been suggested, in accordance with the experimental results.

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1. Introduction

The graft copolymerization of wool fibers with acrylic monomers using chemical initiation is considered to be a powerful method for producing substantial modification in the physical,

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mechanical and morphological properties of the fibers and this affects positively in improving some fiber properties and enduse performance (Ranjbar-Mohammadi et al., 2010, 2013; Wang et al., 2010). Methods of obtaining graft copolymers based on chemical initiation may be divided into a variety of groups (Berlin and Kislenko, 1992). Of these are those based on the reaction of chain transfer through the natural macromolecule, that is, initiation by peroxide compounds and by redox systems of peroxide-metal ion of variable valency. Free radical initiation of grafting, in particular through redox systems, has the following distinctive features (Tsukada et al., 2013; Gawish et al., 2012; Ali and El-Khatib, 2010; Gao and Cranston, 2010; Roy et al., 2009; Moad and Solomon, 2006; Sarac, 1999): (i) it is simple to carry out and no constraints

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in location point of view, (ii) it is inexpensive compared with competitive technologies, (iii) feasibility exists at room temperature and also in the aqueous solution (iv) it has a very short induction period, (v) it possesses a relatively low activation energy. This enables the polymerization to be carried out at low temperature, thereby decreasing the possibility of side reactions which may change the reaction kinetics and the properties of the resulting polymer, (vi) the polymerization is controlled with ease at low temperature and comparatively high molecular weight polymers high yields can be obtained in a very short time, (vii) extent of grafting can be controlled by tuning the reaction variables [viz. mixture composition (monomer/initiator), reaction time, and temperature], (viii) its applicability to the polymerization of a wide range of monomers and thence its ability to provide an unlimited number of copolymers, (ix) its tolerance to a wide range of functional groups and reaction conditions, (x) industrially, redox initiation has widespread application in monomer removal at the end of reaction ('chasing'), and (xi) the redox polymerization further provides direct experimental evidence of the existence of transient radical intermediates generated in the redox reactions, and enables the identification of these radicals as end groups of polymers throwing new light on the reaction mechanisms of redox reactions.

Many authors have made extensive work on the graft copolymerization of vinyl monomers onto wool fibers using a variety of redox initiation systems (Liu et al., 2013; Chauhan and Kaith, 2012; Gao and Kyratzis, 2012; Giri et al., 2003). However, literature survey has revealed that no work has been reported on graft copolymerization of MAA and other acrylic monomers onto wool fabric using a Fe²⁺-wool/sodium perborate (SPB) redox initiation system.

Sodium perborate (SPB) is a white, free-flowing, crystalline compound soluble in water. It exists in the anhydrous, mono-, tri-, and tetra-hydrate forms. Mono- and tetra-hydrate forms are important commercially whereas the tri-hydrate of sodium perborate is not used commercially.

The structure of sodium perborate tetrahydrate was determined by X-ray diffraction in 1961 (Hansson, 1961). The sodium perborate tetrahydrate is reported to form a dimer with a hydrated heterocyclic chair structure sharing six water molecules:



SPB has been found to serve excellently as an efficient oxidizing agent for many of organic compounds (Matteson et al., 1975; McKillop and Koyuncu, 1990; Huestis, 1977; McKillop and Tarbin, 1983). It is also used extensively as a major component in stain removal and deodorizing performance of powder detergent formulations, denture cleaners, automatic dishwasher detergents and various institutional and industrial laundry products (Baker et al., 2001; Zhu et al., 2004). Although SPB is considered as a solid hydrogen peroxide, however, it superiors hydrogen peroxide in many advantages (Zahran, 2006).

The present work describes the optimization of the reaction conditions for grafting of MAA onto wool fabric using SPB as a new initiator by varying the reaction parameters such as different concentrations of SPB, ferrous ion and MAA as well as pH, duration and time of graft copolymerization and studying their effects on the percentages of various polymer criteria including graft yield (%GY), grafting efficiency (%GE), homopolymer formation (%HP) and total conversion (%TC). Each of these parameters was varied one by one keeping other conditions constant in the reaction. Grafting of the acrylic monomers, namely, acrylic acid (AA), methyl acrylate (MA), methyl methacrylate (MMA), ethyl acrylate (EA) and acrylamide (Aam) was also studied under the optimum conditions arrived at for MAA grafting with the aforesaid parameters. This work was further extended to study, individually, the effects of incorporation a reductive transition metal ion activator (e.g. Mn^{2+} and Co^{2+}) and hydroquinone inhibitor on the polymer criteria of MAA grafting onto wool fabric.

Tentative mechanisms including different chemical events that can occur throughout the whole course of polymerization process, are reported.

2. Experimental

2.1. Materials

Scoured and bleached Marino 590 wool fabric (100%) (323 g/m², 2/1 twill) was supplied by Misr Spinning and Weaving Co., El-Mahalla El-Kobra, Egypt. The plain weave fabric [22 picks (weft) and 25 ends (warp)] was used without further purification and cut into samples of known weight (\approx 1 g).

2.2. Chemicals

2.2.1. SPB initiator

Sodium perborate tetrahydrate (SPB), Na BO₃·4H₂O (Aldrich, Germany) was used as received.

2.2.2. Acrylic monomers and their purification

The following monomers were used for the present study:



Figure 1 Graft yield, grafting efficiency, total conversion and homopolymer as a function of SPB concentration: [FAS], 0.5 mmol/L, [MAA], 4%, grafting temperature 70 °C, grafting time, 120 min, pH, 3, M/L, ratio, 1:50.

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