



DEVELOPMENT AND THERMAL CHARACTERIZATION OF CELLULOSE/CLAY NANOCOMPOSITES

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ABSTRACT

Cotton is the most utilized textile fiber and is preferred by consumers over other natural and synthetic fibers for reasons such as comfort and feel [1]. Cotton and other cellulosic fibers, such as kenaf, rami, and flax face competition and shrinking market share due to synthetic fibers, mostly due to product uniformity, performance, and cost. Although cotton is the first choice for apparel, there are markets where natural fibers are unable to compete effectively with synthetic fibers [2]. Regenerated cellulose fibers, such as lyocell and viscose, have been developed to compete with synthetic fibers. Regenerated cellulose fibers may be produced using cotton or other sources of cellulose. One of the major drawbacks to cotton and current regenerated cellulose fibers is flammability. Numerous chemical finishes have been developed to impart various levels of flame resistance to these fibers. These finishes have been successful but somewhat limited in application due to problems with the finish remaining on the material after laundering or product performance regarding wear and abrasion. Due to such limitations, many of these finishes are only used in applications which do not require laundering or expose the material to excessive wear such as draperies and furnishing coverings [3-6].

Over the last 10-15 years research interest has increased in the area of polymer/clay nanocomposites. In most recent research, the polymer matrix has been a synthetic polymer such as polyamides [7], polyimides [8], methacrylates [9, 10], or polystyrene [11]. The typical clay of choice for these composites is montmorillonite. Montmorillonite clay has a large surface area providing sufficient interfacial regions in the nanocomposite, allowing for an enhancement of thermal and tensile properties at low percentages of incorporation into the polymer matrix. The montmorillonite clay has an average length of 2000Å and width of 10Å. Some work has been pursued with natural polymers; however the polymers have been thermoplastics such as cellulose acetate [12, 13]. Additional nanocomposite work with cellulose utilized

cellulose “whiskers” as the reinforcement material and not as the matrix [14-16]. There has been no previously successful work on natural polymers, such as cotton, for the production of polymer/clay nanocomposites.

Several nanocomposite preparation methods were attempted [17]. The method which proved most successful was used for the testing performed in this report. Montmorillonite clay was pretreated with the ammonium salt of dodecylamine according to a previously published procedure [7]. The pretreated montmorillonite clay was stirred rapidly in the solvent 4-Methylmorpholine *N*-oxide (MMNO). After 30 min of stirring, cotton was added to the flask. The cotton/clay/MMNO solution was heated to reflux with continued rapid stirring. The cotton was dissolved approximately 1 hour after reaching reflux. The viscous amber colored solution was removed from heat and reprecipitated into acetonitrile. The material was filtered and washed a second time in acetonitrile. After filtration, the material was washed twice in deionized water. After the final wash and filtration, the samples were collected and dried under vacuum at 120°C.

Powder forms of the nanocomposites suitable for X-ray diffraction studies were produced by precipitation of the hot solution into acetonitrile. The precipitate was filtered and washed three times in DI water to remove residual acetonitrile. The goal of the work was to produce a flame resistant cellulose/clay nanocomposite material that would be suitable for diverse applications, particularly textiles. Textile applications require the production of the material in a fiber form. The manufacture of regenerated cellulose fibers has been well documented for the viscose and lyocell methods [18,19]. The industrial process was mimicked for the production of fibers on a laboratory-scale. The work of Broughton [20] showed that a syringe or HPLC (high performance liquid chromatography) pump could be modified to produce uniform and consistent fibers. This micro-scale system was sufficient to produce approximately 25 grams of fiber per batch. The needle gauge could be varied to produce different diameter fibers, however smaller gauge needles resulted in higher pressures

being needed to extrude the fiber and the syringe pump could be stalled. An 18 gauge needle was

found to be ideal for the production of textile fibers. The 18 gauge needle produced fibers with a nominal diameter of 840 μm .

A variety of physical analyses was performed to characterize the produced materials. X-ray diffraction was performed to verify that the produced materials were true nanocomposites (Figure 1). Thermogravimetric analysis and Differential scanning calorimetry were used to examine changes in the thermal properties of the materials.

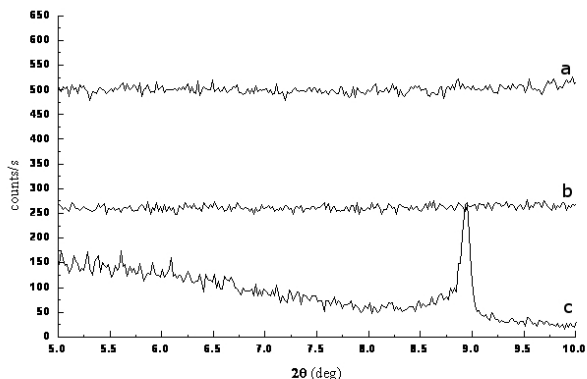


Fig. 1. XRD Patterns of (a) Control Cotton Sample, (b) Nanocomposite with 15% Montmorillonite Filler, and (c) Montmorillonite Clay

A model, based on diffusivity, was developed to help explain the way in which polymer/clay nanocomposites improve the flame retardance of the matrix materials. The theoretical model was validated with experimental results using multiple clay sources of various size characteristics.

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