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Development of a novel cellulose/duck feather composite fibre regenerated in ionic liquid

Rasike De Silva*, Xungai Wang, Nolene Byrne

Institute for Frontier Materials, Deakin University, 75 Pigdons Road, Waurn Ponds, Victoria, 3216, Australia

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ABSTRACT

By blending cellulose and duck feather in the common solvent 1-allyl-3-methylimidazoloium chloride, a regenerated composite fibre has been developed with improved fibres over regenerated cellulose fibres (RCF). The mechanical properties of composite fibre was shown to be better than RCF with a 63.7% improvement in tensile strain. Here, we thoroughly characterise the composite fibre and show that the composite fibre has many advantages over RCFs both from a spinning perspective and as a regenerated fibre.

1. Introduction

Cotton is one of the most important fibre for manufacturing textile yarns among all the textile fibres produced. Its superior properties such as good moisture absorbency, comfortable soft handle and excellent mechanical properties have made the cotton fibre to be a dominant raw material in the textile industry (Wakelyn et al., 2007). However, cotton crops consume large amounts of land and water, which has limited the cotton production over the last few decades (Kotek, 2007; LaNieve, 2007; Wakelyn et al., 2007; Woodings, 2000). This limited cotton supply has not been able to cater the steadily increasing fibre demand driven by the population growth and rapid change in fashion trends (Lenzing.com, 2015). Regenerated cellulose fibres (RCF) are thus becoming important as a substitute for natural cotton fibre to fill this so-called cellulose gap (Kotek, 2007; Lenzing.com, 2015; Wakelyn et al., 2007; Woodings, 2000). RCF are produced by dissolving wood pulp in a solvent followed by coagulation (Kotek, 2007; Lenzing.com, 2015; Woodings, 2000). However, the crystalline structure and the extensive hydrogen bond network hinder the dissolution of cellulose in conventional solvents (Pinkert, Marsh, Pang, & Staiger, 2009). Therefore, efficient and non-toxic solvents and new cellulose regeneration methods are constantly being sought (Heinze & Koschella, 2005).

To date, viscose fibre process is the most commercially available RCF production, in the viscose process; cellulose is derivatised into sodium cellulose xanthate using carbon disulphide (CS_2), enabling cellulose solubility in NaOH. The fibre is then produced through wet spinning from the NaOH solution (Gibril & Yue, 2012; Klemm, Heublein, Fink, & Bohn, 2005; Kotek, 2007). However, this process puts both environmental and human health at a greater risk due to the use of CS_2, a volatile and toxic gas, discharge of harmful waste materials and consumption of large amount of water (Heinze & Liebert, 2001; SIGMA-ALDRICH, 2013; Zhu et al., 2006).

As an alternative for viscose fibre, lyocell fibre was introduced to the textile fibre market in early 1990s (Kotek, 2007). In the lyocell process, cellulose is directly dissolved in *N*-methylmorpholine-*N*-oxide ($NMMO{\cdot}H_20$) and dry-jet spun from the NMMO solution (Fink, Weigel, Purz, & Ganster, 2001; Kotek, 2007; Woodings, 2000). Compared to viscose fibres, lyocell fibres exhibit excellent mechanical properties, in particular in the wet state (Kotek, 2007). Since the $NMMO{\cdot}H_20$ is a solid at room temperature, long processing time at higher temperatures are required in the lyocell process. Moreover, NMMO is thermally unstable and could undergo exothermic runaway reactions at higher temperatures resulting in the formation of by-products due to the degradation of both NMMO and cellulose (Kotek, 2007; Woodings, 2000). The main by-products formed in the process are *N*-methyl-morpholine (NMM), morpholine (M), and chromophores which cause severe discoloration of the solution

* Corresponding author.
E-mail addresses: rdesil@deakin.edu.au, rasiked@rocketmail.com (R. De Silva).

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(Kotek, 2007; Rosenau, Potthast, Sixta, & Kosma, 2001; Woodings, 2000). Therefore, stabilisers have been added to prevent both solvent and cellulose degradation (Rosenau et al., 2001).

Ionic liquids (ILs) are relatively new class of solvents composed entirely of ions and having a melting point below 100 °C (Kokorin, 2011; Rogers & Seddon, 2003). ILs are considered to be environmentally benign and recyclable solvents due to owing very low vapour pressure (Rogers & Seddon, 2003). In recent times, ionic liquids have been shown to be excellent solvents for cellulose dissolution. The dissolved cellulose then can be regenerated in the forms of films or fibres by coagulation in an anti-solvent. Rogers et al. firstly reported cellulose regeneration using ILs (Swatloski, Spear, Holbrey, & Rogers, 2002). Since then, a large number of ILs have been explored for the dissolution of cellulose, largely in the context of biomass processing(Fukaya, Hayashi, Wada, & Ohno, 2008; Hauru, Hummel, Michud, & Sixta, 2014; Hauru, Hummel, Nieminen, Michud, & Sixta, 2016; Heinze & Koschella, 2005; Hermanutz, Meister, & Uerdingen, 2006; Pinkert et al., 2009; Vitz, Erdmenger & Schubert, 2010). The use of ionic liquids to produce RCF is an area of great potential (Hauru et al., 2014; Hermanutz, Gähr, Uerdingen, Meister, & Kosan, 2008; Olsson & Westman, 2013). The benefit of using ILs in RCF processing is that either the wet spinning or dry-jet wet spinning methods can be used (Hauru et al., 2014; Olsson & Westman, 2013). Additionally, the IL can be recycled and reused in the subsequent processing (Cao, Wu, Zhang, Li, Zhang, & He, 2009; Wu, Wang, Wang, Bian, & Li, 2009).

Among a wide range of cellulose dissolving solvents including ILs, only imidazolium based ILs shown to be a **common solvent** type for natural polymers including both cellulose and protein polymers. In general ILs consisting of 1-R_1-3-R_2-imidazolium cation and either the chloride or acetate anion have been used for dissolving cellulose and protein polymers acting as non-derivatised solvents (Goujon, Rajkhowa, Wang & Byrne, 2013; Idris et al., 2013; Li & Wang, 2013; Phillips et al., 2004; Swatloski et al., 2002; Vitz et al., 2010; Xie, Li & Zhang, 2005; Zhang, Wu, Zhang & He, 2005; Zhao et al., 2010). However, cellulose regenerated in these ILs had relatively lower material properties (Cao, Li, Zhang, Zhang & He, 2010; De Silva, Vongsanga, Wang & Byrne, 2015a; De Silva, Wang & Byrne, 2013; Hameed & Guo, 2010) and we have recently shown that these low material properties were directly related to the dissolution temperature as it had a significant impact on the degree of polymerisation (DP) of the regenerated cellulose(De Silva et al., 2015a). As a solution to this, we have recently sought to improve the mechanical properties of the regenerated cellulose via polymer blending using imidazolium based ILs as a common solvent. Polymer blending is a well-known technique used in the polymer industry to develop new materials with improved properties (Wang & Zhang, 2009; Yu, 2008; Yu, Dean & Li, 2006). Duck feathers are a keratin based protein material consist about 90% keratin which contains about 7% cysteine (Idris et al., 2013; Sun, Liu & Liu, 2009; Ullah, Vasanthan, Bressler, Elias & Wu, 2011). It is considered largely to be a waste material in poultry industry (Idris et al., 2013; Zhao et al., 2010). Duck feather is under-utilised as it is a difficult polymer to dissolve due to the tight arrangement of the α-helix and β-sheet in the polypeptide chain (Idris et al., 2013; Sun et al., 2009; Ullah et al., 2011). Additionally, the use of these fibrous waste to regenerate new materials have a greater importance in terms of waste management. The use of duck feather to produce value adding regenerated materials with improved properties has not been studied in detail. In our recent work (De Silva, Vongsanga, Wang & Byrne, 2015b; De Silva et al., 2013) it has been shown that while regenerated duck feather maintains its helical structure, unlike wool which adopts beta structure and becomes very brittle in the regenerated state.

In this study, fibres with different duck feather composition were wet spun from the IL solution AMIMCI. The rheological properties and diffusion kinetics of the blend polymer solutions were investigated and the addition of the duck feather reduced the solution viscosity enabling spinning to be more easily conducted. The mechanical properties of the composite fibres were evaluated as a function of cellulose: duck feather blend ratio. The morphology of the composite fibre surface and the cross sections were investigated using scanning electron microscopy (SEM).

Table 1
Polymer blend solutions prepared for spinning.

Sample name	Polymer blend composition
C100	100% cotton
CDF95	95% cotton; 5% duck feather
CDF90	90% cotton; 10% duck feather
CDF80	80% cotton; 20% duck feather

2. Material & methods

2.1. Ionic liquids used in polymer dissolution

1-Allyl-3-methylimidazolium chloride (AMIMCl > 98%; lot no. 100319.2.1) was purchased from Io-Li-Tec, Germany. The IL was dried under reduced pressure at 85 °C to remove water prior to dissolution. The water content of AMIMCl was measured using a Karl-Fischer coulometer and was determined to be less than 0.8% for all dissolutions.

2.2. Polymers used in fibre regeneration

The cellulose based materials (100% cotton yarns), were provided by Leading Textiles, Melbourne, Australia. These yarns have not undergone pre-treatment, such as scouring or bleaching. The degree of polymerisation (DP) of the starting cellulose material (100% cotton yarn) was measured to be 1260. The DP of the cellulose was determined as the intrinsic viscosity average DP, as described (ASTMStandard, 2013). (The test method is described in the ESI file). Duck feathers were obtained from a duck feather pillow purchased from spotlight, Australia. The cotton and duck feathers were oven dried for at least 24 h at 105 °C prior to dissolution in order to remove the moisture.

2.3. Dissolution of polymer blends

Cellulose/duck feather polymer blend solutions at different ratios were produced in this study. The dissolution process was carried out using a pre-heated heating block with a thermocouple at 100 °C. The polymers were added at increments of 1 wt% under magnetic stirring. After total polymer dissolution of 8 wt%, a clear and viscous cellulose/duck feather blend polymer solutions were obtained. The complete dissolution was observed and verified using a Nikon 80i eclipse polarising light microscope (PLM). A dissolved solution was obtained when no crystallinities in the polymer solution were detected. Table 1 shows the cellulose/duck feather polymer blend solutions prepared for spinning.

2.4. Preparation of regenerated composite fibres

The fibre extrusion was carried out using a simple laboratory set up. The polymer solution was degassed prior to spinning in order to remove any air bubbles contained in the spinning solution. The desired polymer solution (spinning dope) was then transferred into a glass syringe (internal diameter of 10.5 mm) and mounted onto a syringe pump. The temperature of the spinning dope was maintained at 40 °C by a heating jacket attached to the glass syringe. The spinning dope was then extruded into a water bath through