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ORIGINAL PAPER

Mechanical properties of all-cellulose composites from end-of-life textiles

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Abstract
This paper reports the recycling of end-of-life cellulose containing textiles by fabrication of all-cellulose composites (ACCs). Discharged denim fabrics were used as the reinforcement while dissolved cellulose from two different cellulose resources was used as the matrix phase. Virgin cotton fibres and recovered cotton from polyester/cotton (polycotton) waste fabrics were used to form the matrix phase. The process comprises preparing a 6 wt% cellulose solution by dissolving cellulose solution in a ionic liquid, 1-butyl-3-methyl imidazolium acetate ([BMIM][Ac]), this solution acted as a precursor for the matrix component. The denim fabrics were first embedded in the cellulose/IL solution followed by removal of the IL by washing to form the composite. The effect of reuse of the recovered IL by distillation was also investigated. The mechanical properties of the obtained ACCs were determined regarding tensile, impact and flexural properties. Fabricated ACC composite laminates were further characterised regarding structure by scanning electron microscopy.

Keywords Denim fabrics · All-cellulose composites · Fibre/matrix bond · Mechanical properties · Ionic liquid · 1-butyl-3-methyl imidazolium acetate

Introduction

Sustainability is today a trend that is seen everywhere, with no exception for the textiles industry. However, there is a rather significant downside regarding how the textile industry currently operates, namely the huge amount of end-of-life textiles coming along with it. Approximately 73% of the 53 million tonnes of fibres used annually for textile production is landfilled or incinerated, while only 12% is recycled as secondary products [1]. Mechanical recycling of end-of-life textile fabrics into yarns and fabrics was before very common, but due to the low costs for virgin man-made fibres, the current textile material composition diversity, the fibre material quality variations and the high recycling costs this route is not feasible. Another way to decrease the ever-growing pile of textile waste is to repurpose the textile. If a feasible methodology can be found to reuse end-of life textiles in secondary market products by a manufacturing process that requires rather low investment costs, then this could be highly beneficial to counteract the increasing textile waste volumes [2, 3].

A large amount of the textile wastes are fabrics made of cotton [4, 5]. To produce 1 kg of cotton which approximately equals a pair of jeans and one T-shirt, more than 20,000 l of water and a lot of pesticides are used to ensure a good harvest. Denim jeans are among the most popular clothing textiles in the world consumer's demand and a significant part of global cotton production is be applied for denim fabric production [6]. The potential value of cotton textile waste as a source for textile reinforcements for the production of cost-effective and composites has been investigated [7–14]. Structural and mechanically strong composites would an excellent application area for the recycled cotton materials, as composites are durable and light-weight products, with good mechanical properties. All-cellulose composites (ACCs) are monocomponent or single polymer composites, and they are entirely made from cellulose, ideally leading to a homogeneous biocomposite. Since the matrix and the reinforcement are both made from cellulose, and therefore chemically identical, they are fully compatible with each other which allow efficient stress

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transfer and adhesion over their interface [15–17]. Apart from improving the mechanical performance of the final products, the recycling of the composites will be facilitated [17]. ACCs can be obtained through two different processing methods reported in the literature. The first method involves the complete dissolution of a cellulose material with an appropriate solvent followed by adding this solution to fibrous cellulose material which will act as the reinforcement. [18] The second method comprises a selective partial dissolution of a cellulose textile material to form the matrix phase in situ around the remaining undissolved cellulose fibre core [16]. Several researches showed that ACCs mostly have good mechanical characteristics and there are already some studies aimed at optimising the ACC's properties. Researchers have investigated the effect of different cellulose resources both for matrix and reinforcing fibres, solvents, antisolvents, manufacturing processes and conditions, cellulose concentration and etc. [15, 19–21]. But to the best of our knowledge, there are no reports on applying end-of-life textiles for making ACCs.

Many textile products are made from blended yarn fabrics, as the most common is polyester and cotton blends, these are named polycotton. A mechanical separation of waste polyester/cotton blended fabric into its constituents is almost impossible since the polyester and the cellulose fibres are completely mixed in the yarn in the fabrics and cannot be separated mechanically. Chemical treatment would be possible, which involves dissolving or depolymerizing one of the fibre components while maintaining the other. Although several concepts have been reported in the literature, this methodology has also its shortcomings. Dissolving polyester from the blended fabrics is currently not economical and environmentally feasible since the polyester can be dissolved by only a few solvents which are both expensive and toxic [22, 23]. The cellulose component can on the other hand be much easier separated by chemical treatments, by derivatisation or by the use of powerful solvents, such as ionic liquids or N-methylmorpholine N-oxide (NMMO) and as well as acids.

In the context of this study, the initial trials to develop ACCs involving end-of-life denim fabrics and recovered cotton from polycotton waster fabrics were reported. Denim fabric as one of the most widely used materials in the world is high-quality textiles, with good mechanical characteristics, should therefore be good candidates to be recycled as an ACC's reinforcement. The reported experimental procedure involves the preparation of continuous fibre-reinforced ACC laminate specimens by a complete dissolving of virgin cotton and recovered cotton from polyester/cotton (polycotton) waste fabrics in ionic liquids (IL). This dissolved cellulose solution is then added to denim fabrics which acts as reinforcing cellulosic material. The aim of the study is to evaluate the possibility to recycle end-of-life textiles in semi-structural composite for further use in applications such as automotive interior parts, furniture and indoor construction, sports and leisure equipment.

Experimental procedures

Materials

A post-consumer cotton denim fabric was used as a textile reinforcement. Worn out end-of-life fabrics were provided by Texaid (Switzerland). This denim fabric is a cotton twill fabric. The cotton comprises of 94% cellulose, 1.3% protein, 1.2% pectin substance and 0.6% wax [24]. The characteristics of the used denim fabric are shown in Table 1. As the matrix precursor, both virgin and recovered cotton fibres were used. Cotton staple fibres (20 mm) were supplied from Uzbekistan. The end-of-life cotton was made from discharged polycotton bed linen fabrics provided by Textilia AB, Sweden. These cotton/ polyester 50:50 fabrics have been used as bed linens in care centres, hospitals and other institutions. The ionic liquid, 1-butyl-3-methylimidazolium acetate ([BMIM][AC]) with a purity of 96%, was obtained from Sigma Aldrich and used as received. Ethanol of 96% purity was provided by Fisher Scientific. The sulphuric acid (95%) was purchased from VWR International (Sweden).

Methods

Cotton separation from polycotton linen fabrics

The linen fabrics were processed to separate the polyester and cotton fibres by a first treatment with 10 N H_2SO_4, which was followed by a subsequent mechanical treatment. A detailed description of the process is found elsewhere [11]. The treatment gave a heterogeneous mixture consisting of a cotton suspension and solid polyester fibres. The polyester fibres were separated by decantation, and then the remaining cotton suspension was filtered with a Whatman polyamide membrane filter with a pore size of 0.45 μm to separate the cellulose. The cellulose was then dried at room temperature to a powder material before further use. The cellulose powder was then applied as a precursor for the matrix component for ACC composite production. The purity of the cellulose was investigated with FTIR.

Cellulose dissolution

The cotton was dissolved in 200 ± 10 mL [BMIM][AC], at a temperature of 100 °C for 2 h and stirred with an overhead stirrer at a shear rate of 1200 rpm. Two different cellulose concentrations (6 and 9 wt% of cellulose) were prepared in order to find out the best concentration and flow behaviour, and the 6 wt% concentration was then chosen for all further experiments since the solution viscosity was still low enough to do the composite infusion without problems.

Table 1 Cotton denim fabric characteristics

Yarn per 10 cm (tex)		Weave type	Weight, (g/m^2)	Thickness (mm)	Fabric strength (MPa)
Warp 213	Weft 285	Z twill	420	0.66 (0.07)	18.37 (7.28)

Composite laminate manufacturing

The composite laminates were produced by an adapted vacuum infusion technique. As reinforcement, samples of 20 × 20 cm^2 size were cut from the denim fabrics. The samples were dried in an oven at 90 °C until no weight loss was noted to ensure removal of any moisture in order to prevent void formation in the composites. The composite laminates were made by lay up of four layers of denim. Pre-impregnation with the cellulose solution heated to 100 °C was done manually by pouring it on denim fabric. The layers were then stacked and stretched to facilitate air removal, and then they were covered with an at the edges sealed plastic film equipped with a plastic tube to enable compaction by using a vacuum pump. After compaction, the plastic film was removed and the fabric stack was heated in an oven at 95 °C for 90 min. The fabric stack was then placed in a 5 L water bath for 20 h at room temperature. In this coagulation step the dissolved cellulose is regenerated in the water antisolvent into a solid phase, and the reinforcement – matrix interphase is therefore formed. Finally, the laminate was press-dried in a hydraulic press at 110 °C and 0.3 MPa for 8 h. The compositions for the produced ACCs composites are given in Table 2.

Recycling of the ionic liquid

In order to optimise the process to produce the ACCs, recycling of the ionic liquid was done by distillation under vacuum. This was carried out using a rotary evaporator (Laborata 20 eco, Heidolph, Germany) at 80 °C and 102 mbar, until no more water separated from the remaining ionic liquid in the rotary evaporator round bottom flask. The purity of the ionic liquid was verified by IR spectroscopy. The IR spectra of fresh IL (FIL) and recycled IL (RIL) were recorded with an FT-IR spectrometer (Perkin-Elmer spectrometer GXII, USA). The recycled ionic liquid was used in a similar way as the virgin ionic liquid described above.

Table 2 Composition of the produced composites

Sample no.	Reinforcement	Cellulose solution
A1	Denim fabric	Cotton-FIL
A2	Denim fabric	Cotton- RIL
A3	Denim fabric	Recovered cotton-FIL

Characterization of the composites

Mechanical testing Tensile testing was performed in accordance with ISO 527 standard test method with a universal 10KT testing machine supplied by Tinius Olsen Ltd., Salfords, UK. The loading rate was 10 mm/min until the samples broke and the load range was 1 kN.

Flexural testing was conducted on the same machine, and performed in accordance with ISO 14125. The loading rate was 10 mm/min and the load range was 1 kN.

Impact testing was performed according to ISO 179 using a Zwick test instrument with a 5 J swinging arm. The samples were tested in an edgewise manner and un-notched.

Thermal testing Thermogravimetric analysis (TGA) was done using TGA Q500 supplied by TA instrument (New Castle, DE, USA). Samples of approximately 10 mg were heated at 10 °C/min in a nitrogen purge stream from 25 °C to 600 °C.

Scanning electron microscopy (SEM) The fractured surfaces for the composite specimens after 3-point bending test were characterized by field emission scanning electron microscopy (EVO MA10, ZEISS). The surfaces were sputtered with a thin gold layer.

Results and discussion

Recovered cotton from polycotton

Polycotton fabrics used as bed linen were subjected to a controlled separation of the cellulose from the polyester. This was done by the removal of cellulose by static heating in acid solutions and successive mechanical treatments in room-temperature water [25]. Cotton is sensitive to acid, which polyester is very resistant to. Therefore in this approach the separation of cotton and polyester is accomplished by hydrolyzing the cotton while the polyester is maintained physically un-degraded. By this treatment the cotton was efficiently separated from the intact polyester fabric as a powder, see Fig. 1. The recovery rate was high, 98–99%, for both the cotton fibres and the polyester fabrics. The thermal characteristics for the components were characterized by thermogravimetric analysis. The obtained TGA curves of the polycotton fabrics, recovered cotton powder and recovered polyester fabric are presented in Fig. 2. The TGA of polycotton sample shows the onset temperature of cellulose decomposition at 314 °C.

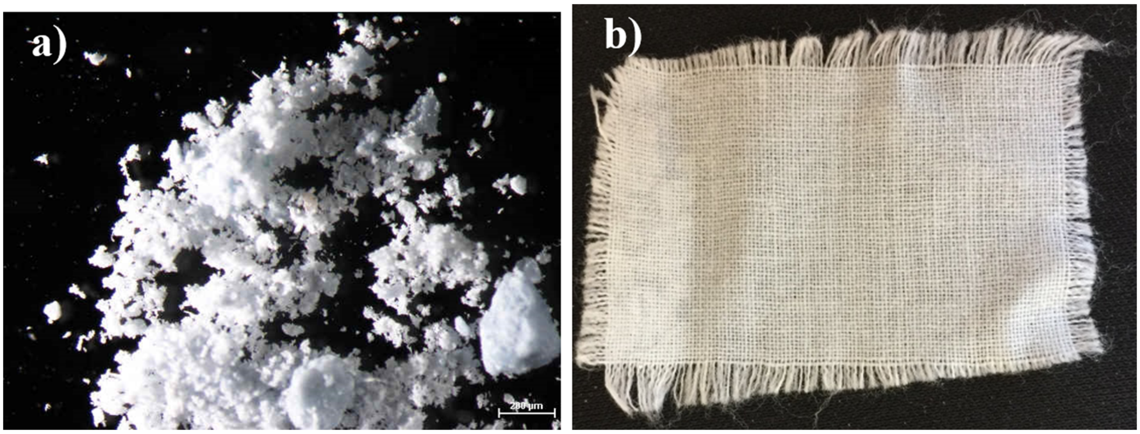


Fig. 1 **a** An optical microscopic image of the recovered cotton powder and **b** an image of the recovered polyester fibres

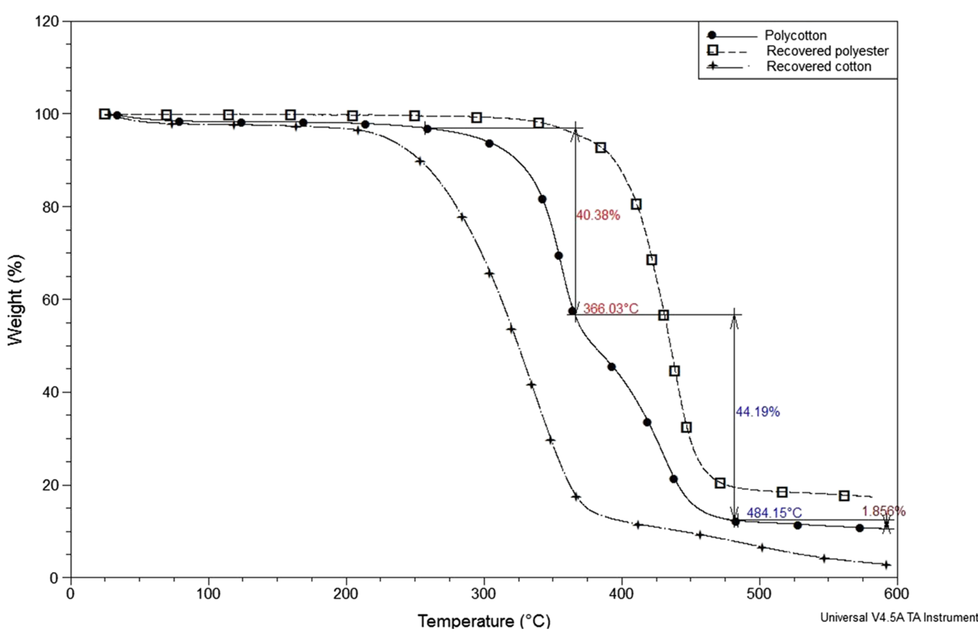
Based on the TGA, the overall decomposition process for the polycotton can be divided into two phases. The first mass loss phase starts at around 260 °C and ends at approximately 366 °C with a mass loss of 40.4%, corresponding to the decomposition of the cotton component in the polycotton fabric, the second mass loss phase continues from there until 484 °C with a mass loss of 44.2%, corresponding to the decomposition of the polyester component. The last step of char pyrolysis yields a mass loss of 1.9%. The thermal degradation up to 600 °C leaves a remaining black residue of 17%. The TGA reveal that the onset of thermal degradation of recovered cotton occurs at a lower temperature than that of the cellulose in untreated polycotton. This reduction of thermal stability can be explained by the presence of sulphate groups, which act as catalysts of the cellulose degradation reactions, as noted in different studies [26–28]. These sulphate groups were formed due to the sulfuric acid treatment used for the cotton separation. The recovered cotton presents only one weight loss process, between 205 and 390 °C and practically no char residue. From TGA measurements, it was confirmed that the recovered cotton is practically pure cellulose.

Figure 3 shows representative FTIR spectra of the virgin cotton staple fibres and recovered cotton from polycotton. The comparison between the spectra displays that all absorbance peaks of both samples were consistent indicating the purity of cellulose obtained from polycotton fabrics.

Recycling of ionic liquid

The IR spectra of the fresh (FIL) and recycled ionic liquids (RIL) are characterized and compared in Fig. 4. The absorbance peaks at 3100–3600 cm^{-1} indicate that small amounts of water remained in RIL and apart from that, all other absorbance peaks of the RIL were consistent with the FIL and no other by-products were seen.

Fig. 2 TGA curves of polycotton, recovered cotton and polyester



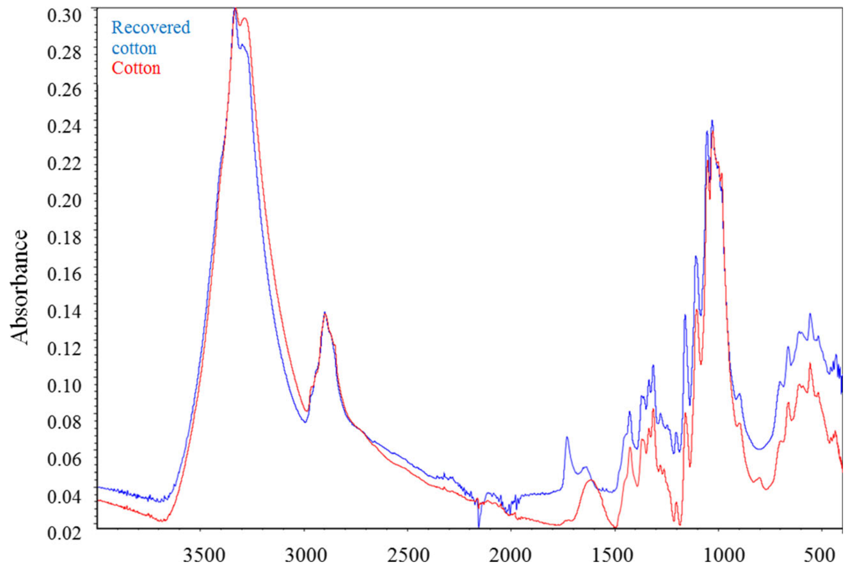


Fig. 3 FTIR spectra of virgin and recovered cotton used as matrix precursor

Mechanical properties of ACC laminates

The influence of different process characteristics, i.e., the use of fresh or recycled ionic liquid and the origin of the cellulose used as the matrix precursor (virgin cotton or recovered cotton from polycotton linens) were investigated. An overview of the tensile modulus and strength of the ACCs compared to the results of other studies is shown in Fig. 5 [4, 10, 15, 20, 29–31]. In brief, the tensile strength for the obtained ACCs in this study ranged from 25 to 29 MPa with an E-modulus from 2.5 to 4.3 GPa which were considerably higher than the values of ACCs reinforced by cotton being reported by Shibata et al. [20]. ACCs were prepared by hot press of cotton fibre/BMIMCl and subsequent annealing. However, the tensile properties of the ACCs prepared by partial dissolution of cotton fibres in LiCl/DMAc [29] were significantly higher than those values obtained here. This could be due to the fact that the mechanical properties of ACCs strongly depend on cellulose resources, cellulose solvents and the different processing conditions including cellulose dissolution and regeneration. Therefore, comparing the properties between different studies of ACCs is challenging [17]. Indeed, study on denim reinforcement is relatively new. Composites are reported of recycled denim fabric added to a polypropylene matrix, manufactured by either hot pressing or hand lay-up. These composites possess 84 wt% of reinforcement [13]. Another study reported the effect of discarded denim and poly(lactic acid) matrix with one to three layers of fabric [12]. Composites were also manufactured from 30% discarded denim fibres in combination with epoxy, polyurethane and

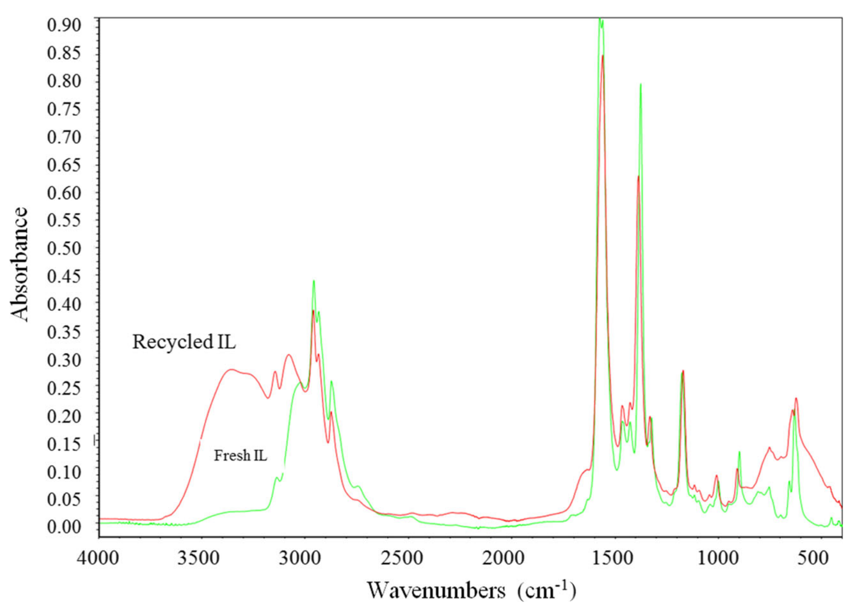


Fig. 4 FTIR spectra of FIL and RIL used in the preparation of ACCs

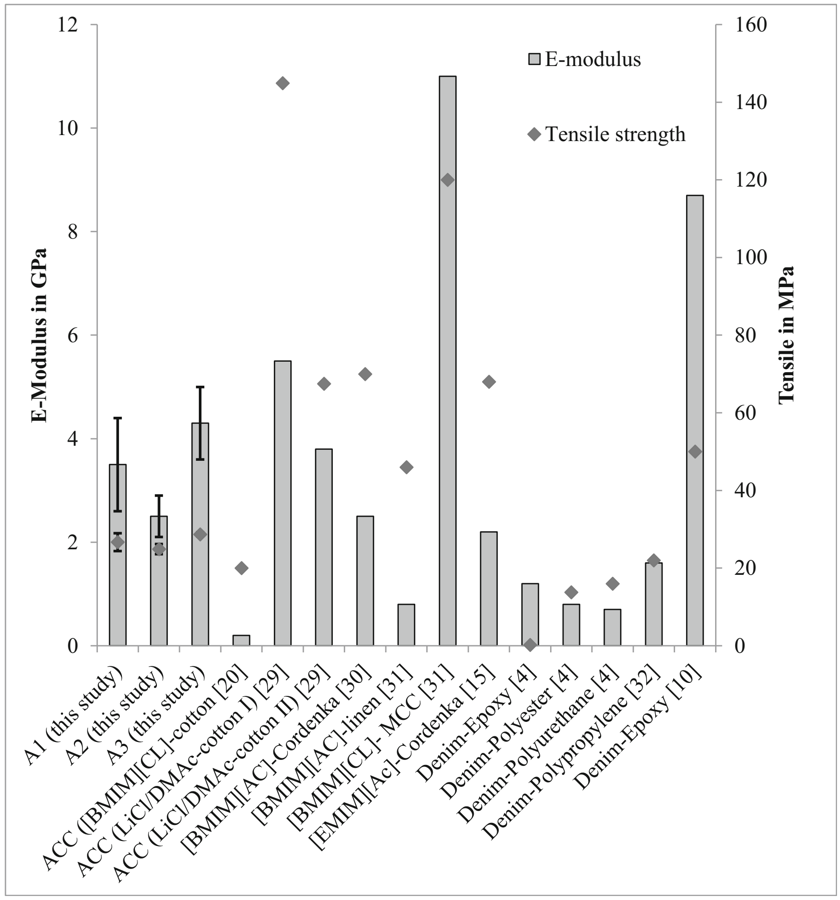


Fig. 5 Mechanical properties of different ACCs and denim-based composites

polyester resin. Compared to the neat resin, all composites have enhanced their mechanical properties [4]. Another study investigated composites from denim scrap in its woven form in combination with a polypropylene resin [32]. In fact, the mechanical performance of our ACCs is higher than that of some other denim-based composites, although still worse than ACCs made with other cellulose raw materials.

As can be seen in Fig. 6 a and b, the flexural modulus was in the range of 1.6–2.1 GPa and the Charpy impact strength reached from 31 to 37 kJ/m^2. When comparing A1 and A2

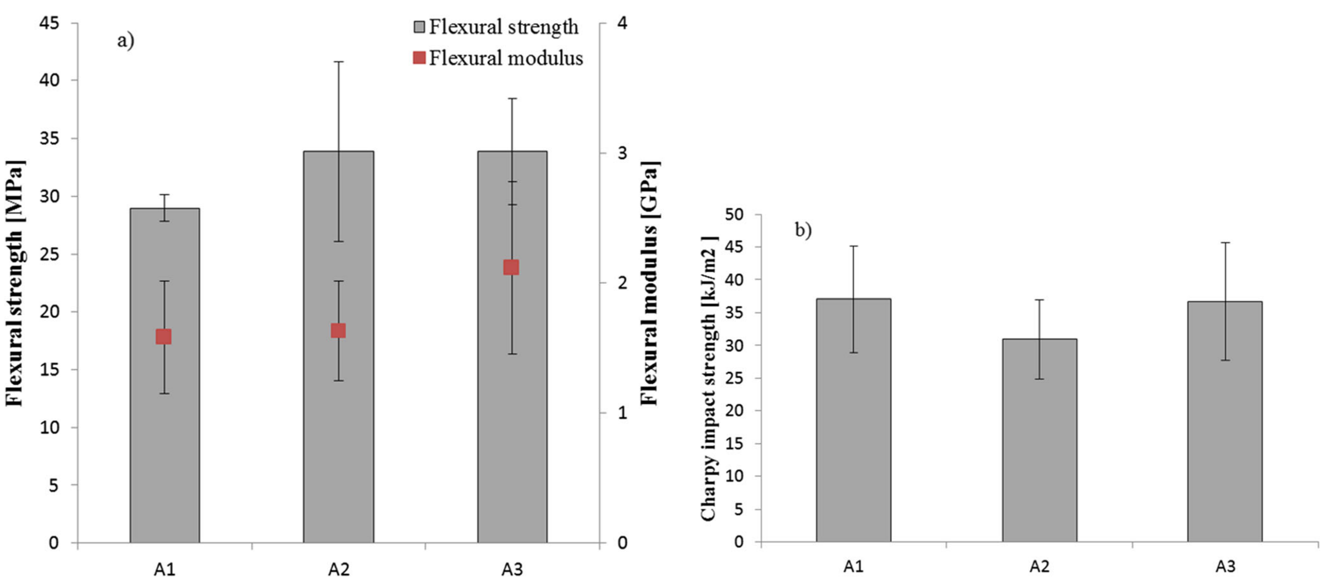


Fig. 6 Flexural (**a**) and impact (**b**) properties of manufactured ACCs

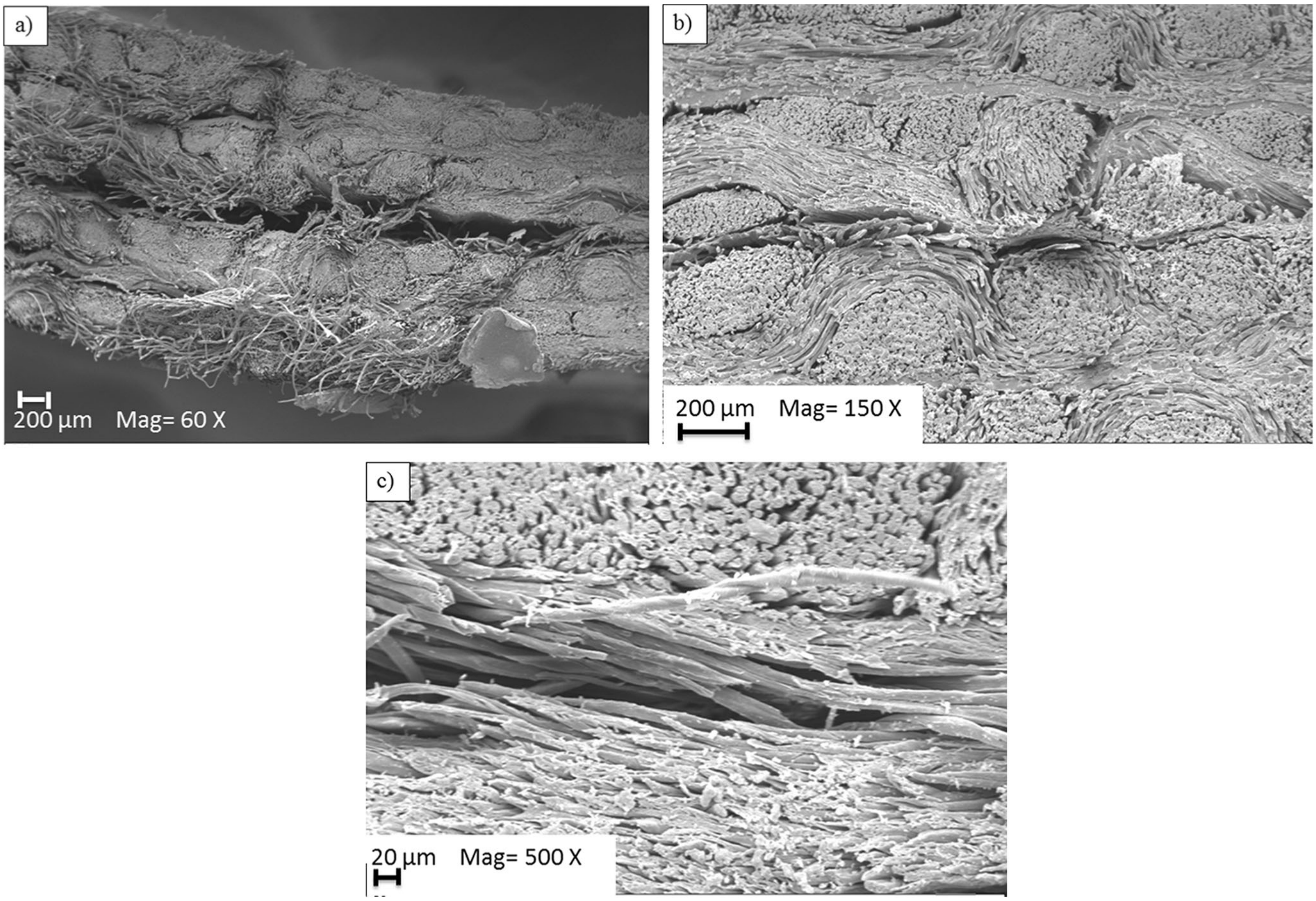


Fig. 7 SEM pictures of the fracture surface of A2 (60, 150 and 500 X magnification)

samples, there is seen no significance in applying recycled ionic liquid (*p* value >0.05), meaning that the solvent recycling for reuse did not seem to impair the cellulose dissolving capacity in ACCs manufacturing. Comparison of different matrix precursors, virgin cotton and the recovered cotton, for A1 and A3 ACCs shows that the incorporation of the recovered cotton did not imply in significant alterations in mechanical properties.

Figure 7 shows the SEM micrographs of the fracture surface of ACCs (A2) after flexural test. As can be seen in the SEM images, consecutive laminar fractures occurred for the denim being caused by the disentanglement of the fibre. On the contrary, the ACCs shows brittle fractures, less and shorter fibre pull-outs which indicates a quite good interfacial adhesion between the cellulose fibers and the cellulose matrix.

SEM micrographs of the flexural fractured surface of ACCs (A2) are shown at different magnifications in Fig. 7. It was observed from fracture surfaces that consecutive laminar fractures occurred for the denim which could either occur because of the disentanglement of the fibre or the absence of a good interfacial bond between the cellulose fibres, as can be seen in Fig. 7 a. It is evident that the cellulose fibres have been surrounded by insufficient matrix to provide a good interface which explains the poor mechanical properties of these ACCs. Furthermore, pulled-out fibres and the corresponding holes are visible in the ACCs (Fig. 7b, c).

Conclusions

Every year tens of million tons of textile waste are generated and mostly disposed in landfills without reuse or recycling. This study aimed at developing a novel and feasible material recycling technique for end-of-life textiles as all-cellulose composites (ACCs). This mono-component composite is completely renewable being composed of only cellulose as both matrix and fibres. Outstanding matrix-fibre compatibility is expected and it will open up new and practical recycling possibilities for the manufactured composite. ACCs were successfully prepared from end-of-life cotton denim woven fabric, different cellulose resources as matrix phase (virgin and recovered cotton from polycotton waste fabric), fresh and recycled [BMIM][AC] (IL). Mechanical testing revealed that there are no clear differences between the values measured for mechanical strength and modulus of the manufactured ACCs from denim/virgin cotton-fresh IL, denim/recovered cotton-fresh IL and denim/virgin cotton-recycled IL. This could be due to the low weight fraction of the cellulose matrix

(~17 wt%) in the final ACC laminates and presumably the denim as cellulose reinforcement strongly influences and dominates the mechanical properties. Although the research completed in the current study has successfully demonstrated that ACCs can be produced by end-of-life fabrics, additional studies on different pretreatments of cellulose for improving the mechanical properties of ACCs, possibility of controlling the crystallinity of cellulose and reducing void fractions are necessary if they are to be used in structural applications. In addition, understanding the effect of shredding process of textile wastes on the mechanical properties of ACCs is of great importance.

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