EXTRACTION OF CELLULOSE FROM DIFFERENT WASTE MATERIALS AS A MEANS TO ILLUSTRATE THE RELEVANCE AND THE POSSIBILITIES OF THE PROCESS OF WASTE UPCYCLING

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ABSTRACT

The aim of this work is illustrating a possible example for explaining the upcycling of waste, hence producing value from it, for possible future use e.g., as viscose in the fashion industry. This has been performed by comparing the results obtained from different cellulose waste, especially yield of the product and its apparent quality. The first objective of the experiments is explaining the chemical procedure to recover cellulose waste for further use. After this, the cellulose extracted is characterized as concerns its morphological structure in terms of dimensions of the fibers obtained. Then, infrared analysis data are compared with those available from previously analyzed cellulose with known composition. Through this experience, the students are introduced to the importance of upcycling in general terms, starting with examples of though they are also presented with the very variable quality of the product obtained from cellulose waste, so to be able to make considerations about the possibility to proceed with the experiment and eventually developing it to an industrial level. *[African Journal of Chemical Education—AJCE 11(1), January 2021]*

INTRODUCTION

In recent years, the extraction of cellulose crystals from waste is a widely followed strategy to increase the purity of the material and its crystallinity in order to confer to it an added value, which would discourage disposing of it just in energy recovery terms. The latter option in fact would also create additional environmental problems, in particular for the treatment by chlorinated agents, typical of bleaching in cellulose-based products, such as those intended for use in the paper sector [1].

As a matter of fact, a number of sources have been used for the extraction of cellulose of high crystallinity to its micrometric or even nanometric dimensions; here, from literature reviews a large number of possible raw materials can be mentioned, some of which are of larger availability and widespread diffusion and therefore easier to be found as waste for disposal [2-3].

For educational purposes, in a school environment, leading down to nanometric dimension can present some obvious difficulties; therefore, the idea was to extract cellulose at a micrometric level, to compare the yield of different waste sources.

The issues, but also the educational content related to this process is multifold: to start with, they include the preparation of the reagent and its relation with the yield obtained in terms of cellulose crystals over weight of the waste used. After this, the consideration for the type of raw material, which is disposed of, is also important, and the comparison, at least qualitative, between cellulose obtained from different materials is of paramount significance.

In this study, the process of cellulose extraction from waste is considered as a wider theme of chemistry education, which would involve a number of sub-themes, including the preparation of different solutions for extraction, the relevant value of the different waste types for the operation, compared against control materials, and the final result, which allows selection depending on the specific type of situation and quality of final material obtained. Evaluation is carried out through the use of different experimental techniques; in particular, some examples of measurements, performed using Fourier transform IR spectroscopy (FTIR) and optical microscopy, are presented.

EXPERIMENTAL

The experiments illustrated were carried out on class formed by 20 students aged 16, of the chemical section of IIS "Fermi-Sacconi-Ceci", in Ascoli Piceno (Italy), whose background already included general and analytical chemistry and were starting to approach organic chemistry. The experience required 3 hours a week on an average, for a period of about 20 weeks, from November to April, together with some theoretical lectures, around once every two weeks.

In particular, the objective of the work was aimed at explaining how cellulose waste can be recovered. The polysaccharide is first recovered from a chemical matrix, including the waste material, a characterization phase then follows, using instrumental chemical analysis, and then the cellulose obtained is compared, according to the waste material used, with respect to two control materials, which are recognized to be made of almost pure cellulose, in practice hydrophilic cotton and microcrystalline cellulose (MCC). The specific waste materials used are orange peels, scrap paper, a worn out tablecloth, and end-of-life jeans trousers. To compare the four waste materials, a weight of 20 (± 2) grams of each waste has been disposed.

The waste has been cut, using scissors when necessary, in small pieces and immersed in 100 ml of Schweizer's reagent, prepared by precipitating copper (II) hydroxide from an aqueous solution of copper sulfate using sodium hydroxide (Figure 1). This is followed by a decantation phase, after which ammonia is added to dissolve the precipitate (Figure 2). The aspect of the four samples with precipitate once immersed in the solution is shown in Figure 3. The four samples collected are examined in a dilute solution of sulfuric acid (5% vol.) at 80°C (Figure 4a), where H_2SO_4 acts as a dehydration catalyst, for cellulose [4]. The excess liquid was removed by pressing the sample between filter papers, the amount of remaining solution was determined by weighing, and the sulfuric acid content was calculated as a percentage to cellulose weight. After this, the formation of a gel-like cellulose suspension was obtained with a 10% vol. glycerol suspension (Figure 4b). The following characterization phase was performed by first measuring the length and diameter of the cellulose segments obtained, from observations under the optical microscope. Also, FTIR-ATR "Spectrum Two" Perkin Elmer spectroscope has been used to analyze the presence of functional groups typical of cellulose.

RESULTS AND DISCUSSION

A number of observations, developed here below, can summarize the educational content of the experience.

First, a theoretical part of the experience was carried out, in which the students familiarized themselves with the issue of extracting microcellulose from different kinds of waste and with the problem of different yielding of the process, which makes it practicable, in economical and environmental terms. Although only qualitative evaluations of the yielding have been carried out, its dependence on the aspect of the raw material has been clearly identified, and, as for an order of magnitude, it has been suggested that the material extracted, though pure and crystalline, does not exceed a few percent of the raw one.

The educational questions that the experience poses are in particular:

- Use of “control” samples (from microcellulose acquired on market) and comparison with the experimental ones (from orange peel, tablecloth, paper waste, and end-of-life jeans)
- Chemical methodology (preparation of the Schweitzer’s reagent, decantation, yielding of the transformation process)
- Morphological characteristics of the microcellulose obtained, as reported from optical microscopy examination, as reported in Figure 5, with measurement of the length and diameter, and observation of the thickening nodes, presence of loose parts and straightness of the fiber. This study has been performed in view of possible insertion in polymer matrices, with the idea to create polymer nanocomposites, on which some indications have been offered to the students, such as those reported in [5]
- Interpretation of FTIR spectra with respect to the material obtained (purity of the cellulose, presence of non-polysaccharide materials), which are depicted in Figure 6.

Following this, the infrared analysis (FTIR-ATR) was carried out to characterize the obtained cellulose and compared with data from MCC and hydrophilic cotton available from previous analysis. In Figure 6, the spectra of the residue after centrifugation and washing with de-ionized water are reported, with the aim not to alter the composition of the sample system.

In FTIR spectra, the characteristic peaks of a carbohydrate are observed: the one around 3300 cm^{-1} relative to the stretching of hydroxyl group -OH, at around 2900 cm^{-1} the peaks of $-CH_2$ and -CH groups stretching, while at 1635-1655 cm^{-1} some peaks characteristics of -CH, $-CH_2$ bending and a peak at 1000-1050 cm^{-1} defined by the stretching of the single bond C-O. These are the main peaks observable, from the large literature available on FTIR analysis of carbohydrates, for example in [6].

In general terms, from FTIR the extracted residue has approximately the same functional characteristics. However, it needs also to be noticed that in the sample originated from table cloth a further peak has been observed, on which the students' attention has been drawn. This peak at circa 1740 cm^{-1} is characteristic of a carbonyl C=O stretching mode, this suggests that in the condition used a partial extraction of a small amount of a polyester fiber has also been carried out. This indicated that the tablecloth was made in a typical cotton/polyester (normally Polyethylene terephthalate, or PET) blend, on which equally the attention has been reported, since FTIR spectroscopy appears as an essential tool to start evaluating the effectiveness of recycling this kind of textiles [6].

This process exposes the difficulty in obtaining an effective upcycling (i.e., revalorization) of the material, as defined in [7], on which some indications have been given to the students in the theoretical lectures. This issue is significant in this case especially for the large difference in the yield and quality of cellulose obtained from the waste material. This would in some cases discourage the recovery of the material in this form, yet this choice is once again part of the evaluation to be carried out. Future developments could involve the modification of cellulose molecules to functionalize them, which may be the objective of further educational projects.

CONCLUSIONS

The experience reported possessed an educational content both in general terms, as to illustrate how working with cellulosic waste can practically lead to the extraction of very pure and oriented microcellulose fibers. This included laboratory experiments and measurements and enabled practicing, other than with preparation of solutions and further processing, also with different techniques of wide use in chemistry practice, such as microscopy and IR spectroscopy.

Some limitations to this approach are recognized to the need to be concentrated in a short period, due to the time possibly dedicated during the school year. On the other side, they can be resumed at a later stage during student education, for example concentrating on film production including microcellulose extracted from waste.

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FIGURES

Figure 1 Preparation of Schweizer's reagent

Figure 2 Decantation phase

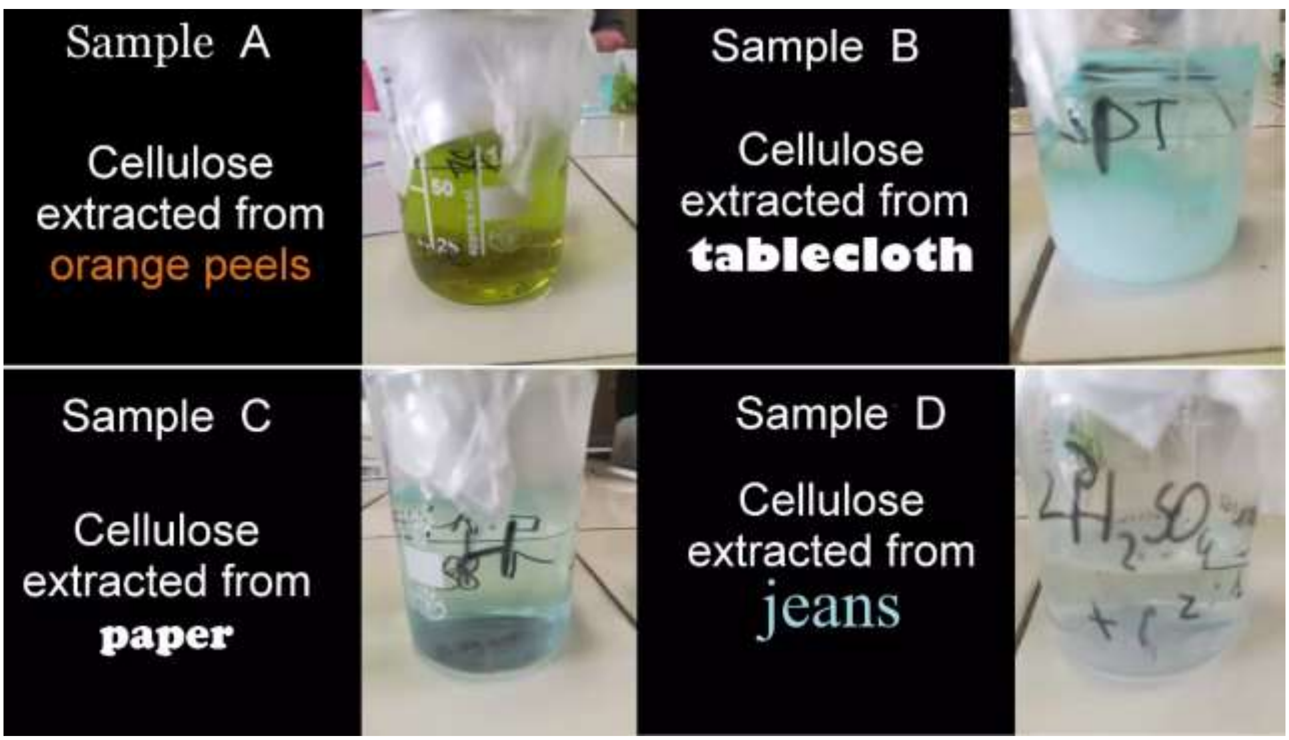

Figure 3 Samples examined

Figure 4 Examination of samples using sulfuric acid and glycerin

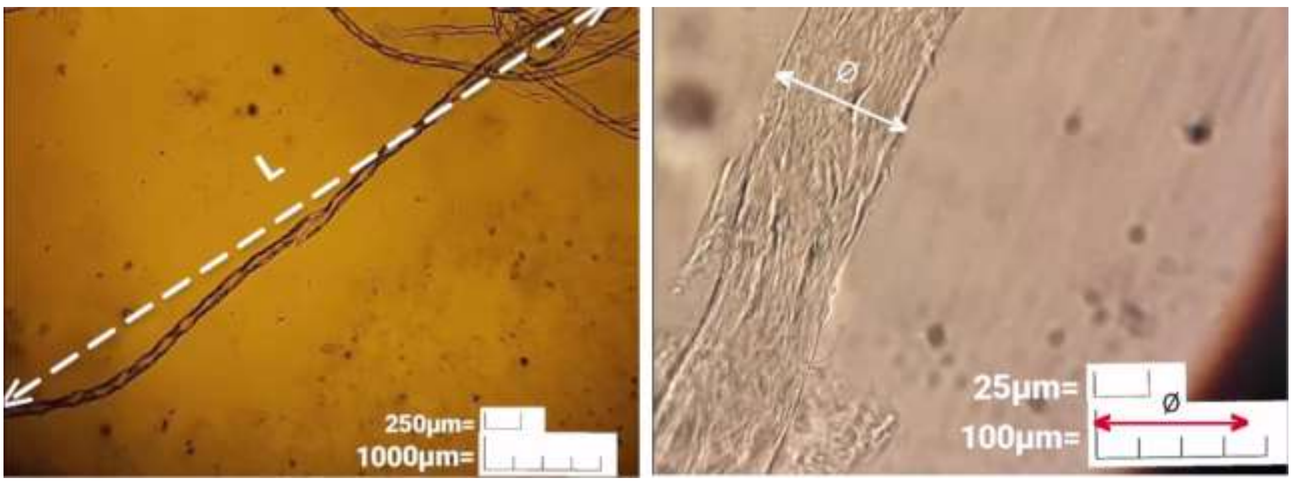

Figure 5 Microscopic observation of microcellulose fibers extracted

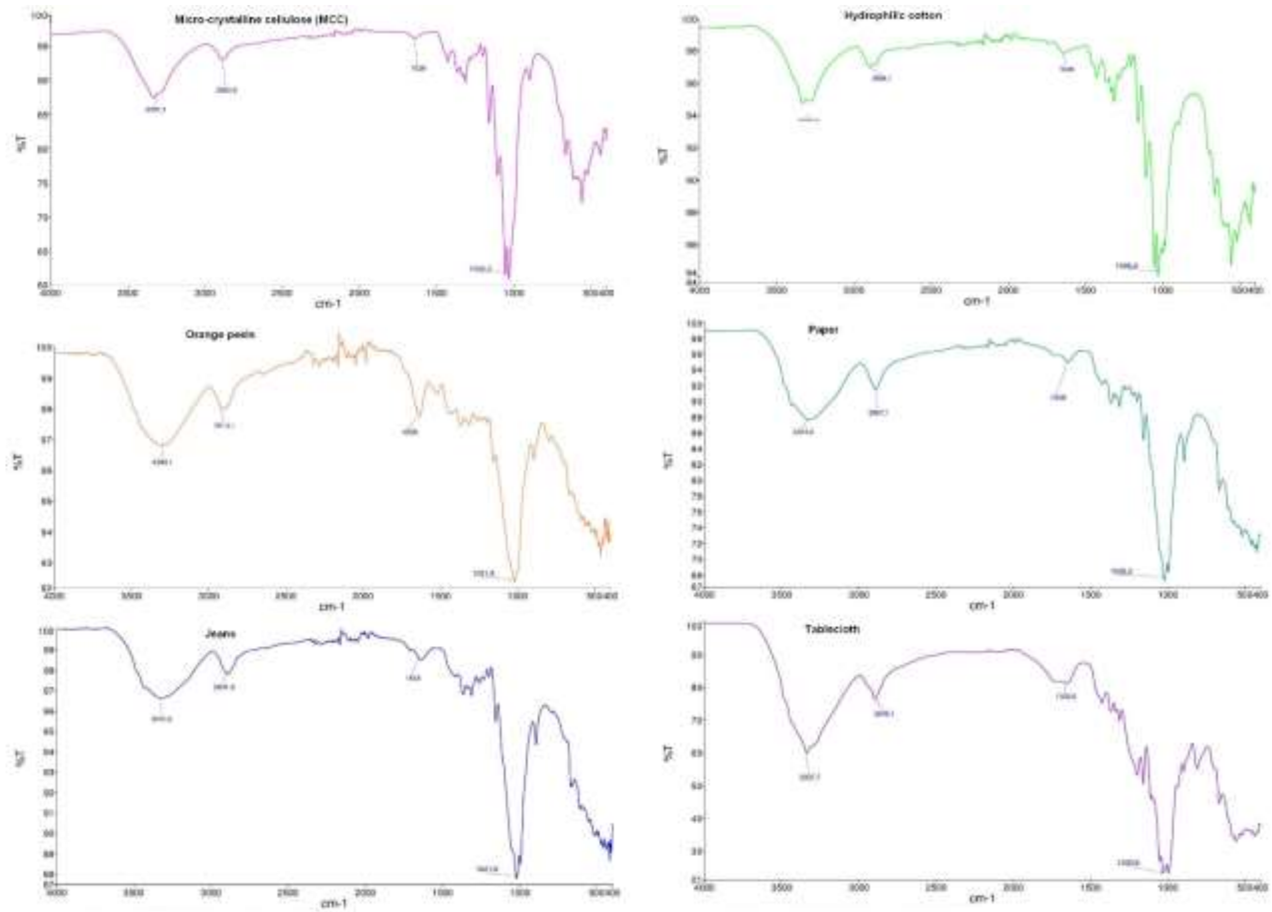

Figure 6 FTIR spectra of the different types of microcellulose