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Revalorization of Rice Husk Waste as a Source of Cellulose and Silica

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Abstract: Cellulose and silica were obtained from rice husk by a multi-step procedure. Three different strategies including different alkaline and bleaching steps were evaluated by Fourier transform infrared spectroscopy (FTIR) in order to choose the sequence that combines the better conditions to obtain both subproducts simultaneously. The effects of the different steps on the structure and morphology of original material treated by the selected procedure were analyzed by X-ray diffraction (XRD), thermogravimetric analysis (TGA), and scanning electron microscopy (SEM). The results suggest that it is possible to obtain both by-products with a simple procedure, giving an added value to this waste.

Keywords: Rice husk, Chemical treatment, Cellulose, Thermal properties, Morphology, Silica

Introduction

Cellulose is the most abundant natural polymer. The most commercially exploited natural resource containing cellulose is wood [1] but cellulose is the main component of several other well employed natural fibers such as banana, coconut husk fibers, cotton, flax, hemp, jute, sisal fibers, and waste such as rice husk [2-6]. Cellulose in these natural resources is embedded in non-cellulosic matrix mainly formed by hemicellulose and lignin.

RH is one of the major agricultural residues generated during the rice milling process [7]. The global rice production stands at around 466 million tons in 2010/2011 (FAO, 2010) [7,8]. About 23 % of this amount is used as a bedding material for animals and discarded in land fillings or simply burned in the fields leading to air and soil pollution [9,10]. Approximate composition of RH is cellulose (25 to 35 %), hemicelluloses (18 to 21 %), lignin (26-31 %), silica (15-17 %), and soluble materials (2-5 %) [11], which makes it an attractive source for the extraction of silica and cellulose.

Many previous studies have been focused on the production of amorphous silica from this residue. Silica obtained from rice husk has found an increasing market in industries, ceramics, electronics, catalysis, pharmaceutics, dental materials, and others [8-10]. Silica is mainly accumulated on the outer epidermis of rice husk [12]. The most common procedure to extract silica is calcination [10,13,14], where high temperature causes the complete loss of organic compounds. Johar *et al.* [15] isolated cellulose from rice husk using classical chemical alkali and bleaching treatments. In this work, however, the extraction of silica was not reported in any step of the procedure. Recently, we proposed a new strategy based on chemical treatments reported in the literature that allows the separation of silica and cellulose simultaneously from rice husk [6,11]. Silica can be isolated by a combination of alkaline treatments followed by precipitation with acid (mainly hydrochloric acid, HCl) [6]. After that cellulose was isolated from organic residue by means of a multistep procedure consisting of alkali and bleaching treatment steps.

The aim of this work is to analyze different sequences of the chemical treatments studied in previous works [6,11,15] in order to concurrently extract cellulose and silica from rice husk using alkali and bleaching treatments. Then, the chemical and physical characteristics of the cellulose obtained will be compared with that of previous studies. The effects of each chemical treatment on the chemical structure, morphology, and thermal properties of the original material were studied by scanning electron microscopy (SEM), thermogravimetric analysis (TGA), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR).

Experimental

Materials

RH was obtained as a residue of rice industries of Entre Ríos (Argentina). Rice husk was extensively washed with distilled water to remove dust and other impurities. This operation was carried out several times at room temperature under vigorous stirring. After successive washings, rice husk was dried in an air-circulated oven at 80±1 °C and then crushed for 10 min using a ball mill (Pulverisette 7, Fritsh, Alemania). The final moisture content of the material was

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about 4-5 wt%. After that, the rice husk powder passing 100 meshes sieve was stored in hermetic plastic containers in order to prevent microbial attack (i.e. fungi). This sample was named S0.

The reagents used for the chemical treatments were: hydrochloric acid (HCl, Cicarelli, Argentina); sodium hydroxide (NaOH, Anedra, Argentina); potassium hydroxide (KOH, Anedra, Argentina); sodium chlorite ($NaClO_2$, FlukaChemie, Germany); sodium bisulfite ($NaHSO_3$, Barker, USA); and buffer solution pH 4 (Anedra, Argentina).

Chemical Procedures for Cellulose and Silica Extraction

Rice husk powder (S0) was submitted to different types of chemical treatments (3 procedures) in order to obtain cellulose and silica. Procedure I was based on our previous studies [6,11]. Procedure II was proposed as an alternative of procedure I changing the treatments sequences looking for the concurrently extraction of high purity cellulose and silica. Procedure III was based on previous studies found in the literature for rice husk [15]. Figure 1 presents the three schemes of chemical separation procedures that have been carried out.

The following paragraphs describe the details of each treatment step.

Procedure I

Rice husk powder (S0) was stirred with 3 % (w/v) of KOH at a weight ratio of 1:12 and boiled for 30 min, and then the mixture was left overnight. The filtrate was washed twice with distilled water, and 10 % (v/v) HCl was added (100 m*l*). In this last step, the organic part rich in cellulose (SI-Or1) was retained in the filter while the silica (SI-In1) precipitated in the liquor [11,16]. After that, the organic part was treated with 0.7 % (w/v) of sodium chlorite at a ratio of 1:50 g solid/m*l* liquor at pH 4 and kept boiling for 2 h. The remaining solid was first treated with 5 % (w/v) sodium bisulfite solution at room temperature for 1 h using a solid to liquor ratio of 1 g/50 m*l* and then washed with distilled water and dried at 80 °C in vacuum oven (SI-Or2). Finally, the organic part was treated with 17.5 % (w/v) of NaOH solution, at room temperature for 8 h using a solid to liquor ratio of 1 g/50 m*l*, washed, and dried at 80±1 °C (SI-Or3).

On the other hand, the liquor rich in silica (SI-In1) was centrifuged, washed with distilled water several times and lyophilized obtaining a dried powder (SI-In2).

Procedure II

Rice husk powder (S0) was treated with 5 % (w/v) of $NaClO_2$ at a ratio of 1:75 g solid/m*l* liquor at pH 4 and kept boiling for 3 h. After that, it was filtered and washed several times with distilled water until neutral pH was reached. The remaining solid was first treated with 20 % (w/v) $NaHSO_3$ solution at room temperature for 30 min using a solid to liquor ratio of 1 g/50 m*l*. Then it was washed with distilled water until neutral pH was reached and dried at 80 °C in vacuum oven (SII-1). After that, SII-1 was stirred with 3 %

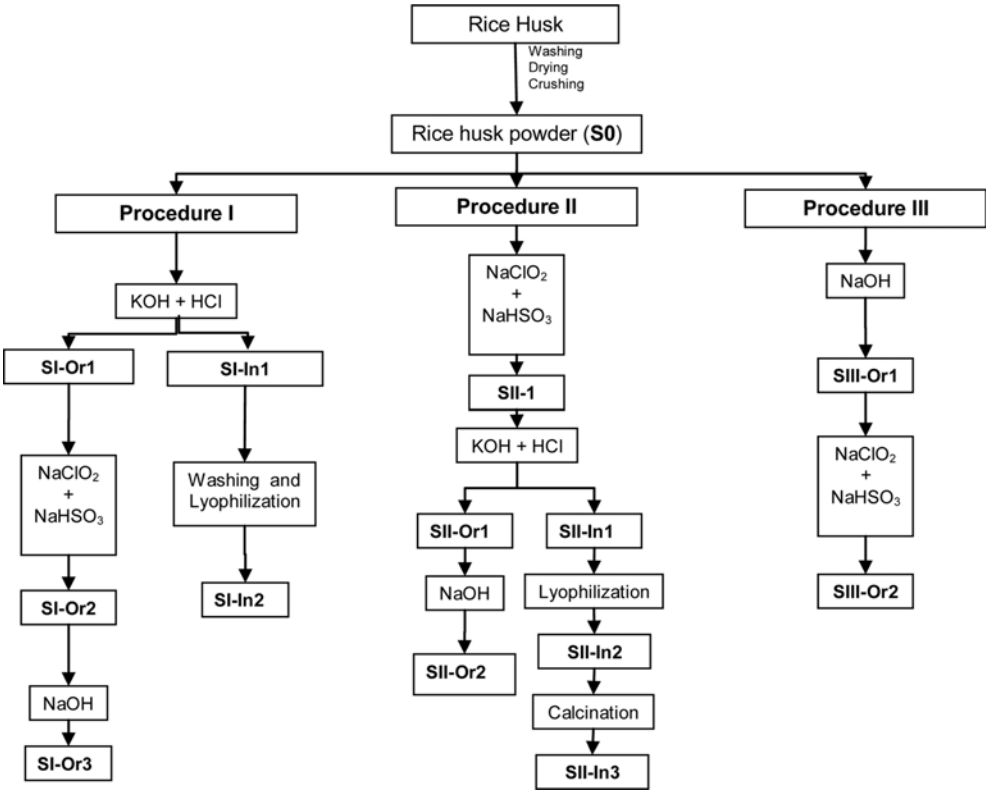

Figure 1. Scheme for isolation of cellulose and silica by three different chemical procedures.

(w/v) KOH at a weight ratio of 1:12 and boiled for 30 min. The mixture was left overnight. The filtrate was washed twice with distilled water, and 10 % (v/v) HCl was added (100 m*l*). In this last step cellulose (SII-Or1) was retained in the filter while silica (SII-In1) precipitated in the liquor. SII-Or2 sample was washed with distilled water until neutral pH and dried again as in the previous step. After that, SII-Or1 was treated with 17.5 % (w/v) of NaOH solution, at room temperature for 8 h using a solid to liquor ratio of 1 g/50 m*l*, washed until neutral pH and dried again as in the previous steps (SII-Or2). On the other hand, the solution rich in silica (SII-In1) was washed with distilled water several times until neutral pH and then it was frozen and lyophilized obtaining a dried powder (SII-In2). Finally the last sample was calcinated in an oven under air atmosphere at 600 °C for 2 h (SII-In3) [17].

Procedure III

Rice husk powder (S0) was treated with 17.5 % (w/v) of NaOH solution, at room temperature for 8 h using a solid to liquor ratio of 1 g/50 m*l*, and washed several times with distilled water until neutral pH was reached. The filtrate was dried at 80±1 °C in an air-circulated oven until constant weight was reached (SIII-Or1). Then, it was treated with 5 % (w/v) of $NaClO_2$ at a ratio of 1:75 g solid/m*l* liquor at pH 4 and kept boiling for 3 h. After that, it was filtered and washed several times with distilled water until neutral pH was reached. The remaining solid was first treated with 20 % (w/v) of $NaHSO_3$ solution at room temperature for 30 min using a solid to liquor ratio of 1 g/50 m*l*, washed with distilled water and dried at 80±1 °C in an air-circulated oven until constant weight was reached (SIII-Or2).

Characterization Methods

FTIR

FTIR spectra of initial rice husk and the products obtained by each treatment step were carried out in transmission mode by using a Mattson Genesis II spectrometer. Samples were pressed with KBr and scanned from 4000 cm^{-1} to 400 cm^{-1} at a resolution of 2 cm^{-1}. 16 scans were performed over each sample. Tests were carried out at room temperature.

XRD

X-ray patterns were obtained from a PW1710 diffractometer equipped with an X-ray generator (λ=0.1539 nm) in the range of 2θ from 5 ° to 60 ° at 1 °/min. From the obtained patterns, it was possible to estimate the crystallinity index of the cellulose based materials as follows [18]:

$$I_c\ (\%) = \frac{(I_{200} - I_{am})}{I_{200}} \times 100 \quad (1)$$

where I_{200} is the counter reading at peak intensity at a 2θ angle close to 22 ° (which is separated into two peaks at 22 ° and 20 ° as cellulose is transformed from cellulose I to cellulose II) representing crystalline material and I_{am} is the intensity of the peak at 2θ angle close to 18 ° representing the amorphous part of the material.

The relative quantity of cellulose II respect to cellulose I ($I_{II\text{-}I}$) was calculated by the ratio of the intensities of the peaks centered at 2θ=20 ° (I_{20}) and 22 ° (I_{22}), whose represent the crystalline part of the material:

$$I_{II\text{-}I}\ (\%) = \frac{I_{20}}{I_{20} + I_{22}} \times 100 \quad (2)$$

TGA

Thermogravimetric analysis (TGA) was used as a tool to determine the initial relative composition and the effect of chemical treatments on the composition of rice husk. TGA measurements were carried out in a Shimadzu TGA-50 (Japan). The samples (5 to 7 mg) were heated from 25 °C to 750 °C at a heating rate of 10 °C/min under nitrogen and air atmospheres (20 m*l*/min). Data obtained from TGA experiments were transformed in ASCII format and analyzed using a statistics computer program. The initial decomposition temperature (IDT) was measured as the onset of weight loss taken at 1 % [17]. Multiple parameters were calculated from derivative thermogravimetric analysis (DTGA): T_{sh} is the mean value of the range of temperatures of the shoulder located on the left of the main peak; T_α the temperature of the maximum of the main peak and T_β is the temperature of the maximum of the secondary peak located on the right of the main peak [6].

SEM

SEM observations of the products obtained by each treatment step were performed with a JEOL JSM-6460 LV (Tokyo, Japan) microscope at acceleration voltage of 15 kV. Prior to the observation, the surfaces were sputter-coated with a gold layer of about 100 Å to avoid charging under the electron beam.

Results and Discussion

Selection of the Optimal Experimental Procedure by Means of FTIR Analysis

The chemical composition of the rice husk and products obtained in each treatment step of the different selected procedures was analyzed by FTIR. The FTIR allows characterizing the chemical structure by identifying the functional groups present in each sample. The main organic components of rice husk show characteristic bands associated with alkanes, esters, aromatics, ketones, and alcohols with different functional groups containing oxygen [11]. Hemicelluloses bands can be easily identified at 1732 cm^{-1}, which corresponds to vibration of acetyl and uronic ester groups [19-21]. The main peaks of cellulose can be observed at 1635 cm^{-1} (OH bending of adsorbed water), 1430 cm^{-1} (strain CH_2), 1372 and 1270 cm^{-1} (bending CH) [4,22,23]. The vibrations of the aromatic rings of lignin appear at 1594 and 1509 cm^{-1} [4,6]. The inorganic part of rice husk can be observed in two characteristic bands, associated with Si-O-Si bonds, centered at 799 and 465 cm^{-1}

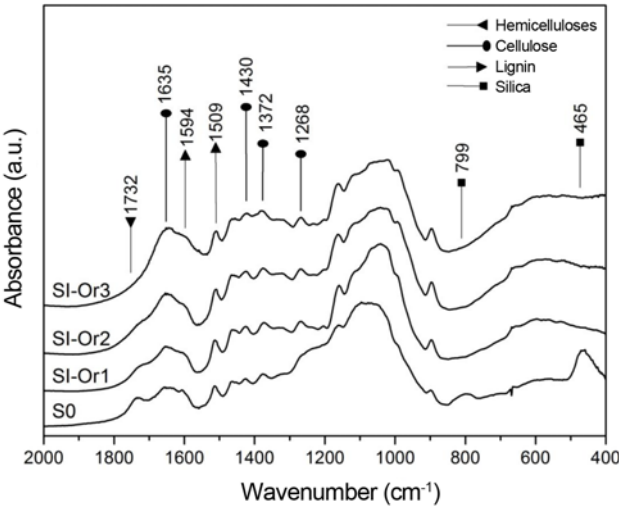

Figure 2. Spectra of rice husk and organic sub products procedure I.

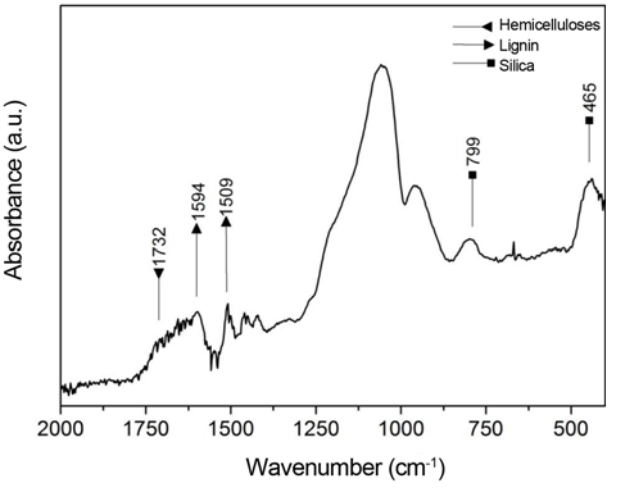

Figure 3. Spectra of inorganic product from procedure I.

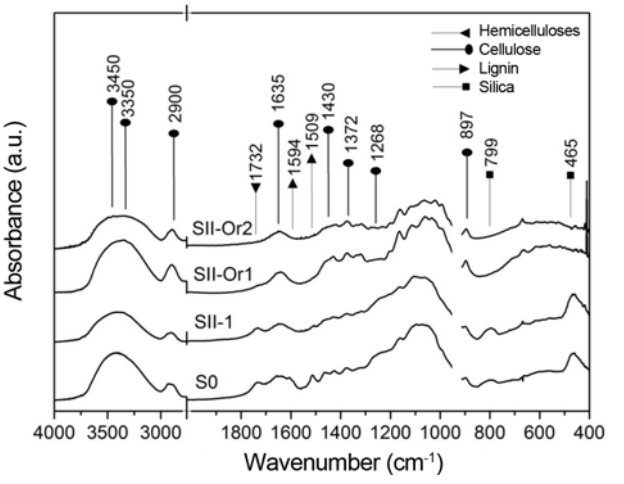

Figure 4. Spectra of rice husk and organic sub products procedure II.

[6,11,21]. The previously mentioned bands were used in the present work in order to evaluate the effectiveness of each chemical treatment on the preferential removal of different components. In all cases a break in the X-scale of Figures from 2000 cm^{-1} to 2800 cm^{-1} was inserted due to the fact that no bands were present in this range.

Figure 2 shows the FTIR spectra of rice husk and organic sub-products obtained from procedure I. After the first step, (SI-Or1), the bands centered at 799 cm^{-1} and 465 cm^{-1} related to Si-O-Si bonds disappeared which confirms that the silica was completely removed by the alkaline treatment of the rice husk. In addition, the intensity of peak centered at 1732 cm^{-1} decreased indicating the partial elimination of hemicellulose [19-22]. The color of this sample remained dark which was related to the presence of lignin also verified by the peaks at 1509 and 1594 cm^{-1}. After the treatment steps with $NaClO_2$+$NaHSO_3$, (SI-Or2), and with NaOH (SI-Or3), the peak attributed to the hemicelluloses, centered at 1732 cm^{-1}, completely disappeared whereas the peaks centered at 1509 and 1594 cm^{-1} were still present, indicating that the conditions used for the procedure I were not effective to achieve complete removal of lignin.

The peaks centered at 465 and 799 cm^{-1}, which corresponds to the silica, can be clearly identified in the Figure 3. It was also found that even when the washing and rinsing process were repeated several times (SI-In2), residues of lignin and hemicelluloses were still present (bands at 1509, 1594, and 1732 cm^{-1}) in the liquor. It was possible to conclude that this treatment was not effective in separating pure silica and cellulose fractions.

Based on the previous results, a new strategy (procedure II) was designed in order to optimize the extraction of lignin and hemicellulose from raw materials and to obtain pure cellulose and silica. The order of the treatment steps was reversed with respect to procedure I starting with the $NaClO_2$+$NaHSO_3$ treatment and following by the KOH+HCl treatment (see Figure 1). In addition, $NaClO_2$ concentration was increased from 0.7 to 5.0 % (w/v) with the aim of improving the extraction of lignin. FTIR spectra obtained by applying procedure II are shown in Figure 4.

After the first step, (SII-1), the peaks centered at 1509 and 1594 cm^{-1} disappeared which indicates that lignin was completely extracted. Peaks related to hemicellulose (1732 cm^{-1}) and silica (465 and 799 cm^{-1}), can be still observed in the spectra. After the KOH+HCl step, (SII-Or1) hemicellulose proportion in the sample was significantly reduced whereas the silica was completely extracted from the organic part. Finally, SII-Or2 sample only shows the peaks associated with cellulose because in this last step, the residue of hemicellulose was completely removed, by means of NaOH treatment. FTIR spectrum of lyophilized liquor rich in silica (SII-In2) is shown in Figure 5. The silica bands, centered at 799 and 465 cm^{-1}, and one of characteristic peaks of

cellulose, centered at 1635 cm^{-1}, can be observed in that phase. The presence of cellulose in the liquor rich in silica indicates that a small amount of cellulose was partially dragged during the washing and filtering step with HCl. Finally, this sample was calcinated at 600 °C for 2 h obtaining pure silica (SII-In3 in Figure 5).

Figure 6 show the FTIR spectra of rice husk and organic subproduct obtained from procedure III. It can be noted that the NaOH treatment was very efficient in removing hemicellulose and silica, because no peaks were observed at 1732, 799, and 465 cm^{-1} (SIII-Or1). However, it was not possible to separate silica by precipitation as in the previous procedures. $NaClO_2$+$NaHSO_3$ treatment (SIII-Or2) was effective to obtain pure cellulose as lignin peaks (1509 a 1594 cm^{-1}) completely disappeared.

From all previous results, it can be concluded that procedure II meets the best conditions in order to obtain cellulose and silica simultaneously. Applying this procedure, 15.5 g of cellulose and 15 g of silica were obtained from 100 g of rice husk. In the following section the results of the characterization by SEM, TGA, and DRX of samples obtained by the selected procedure will be also described.

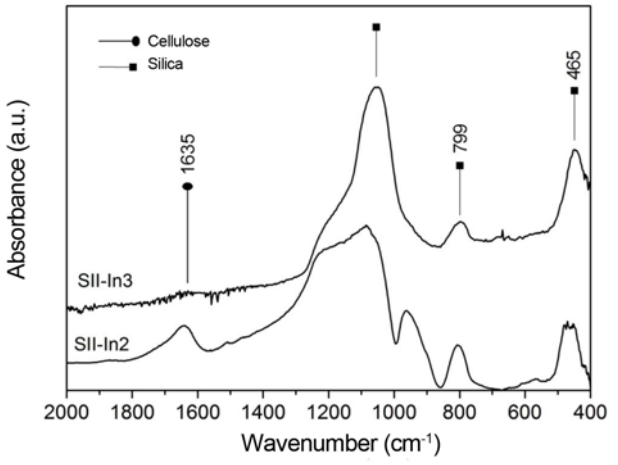

Figure 5. Spectra of inorganic product from procedure II.

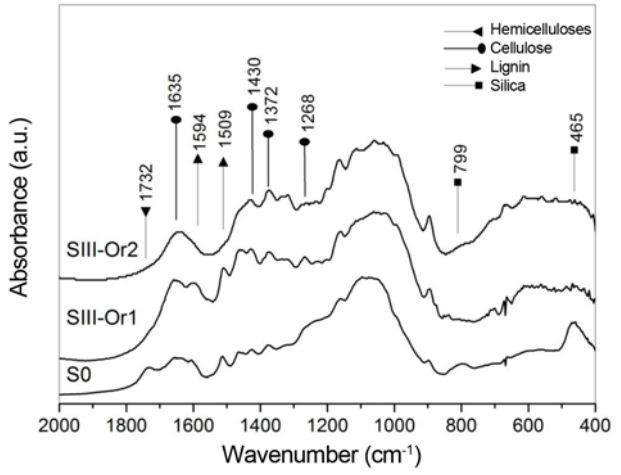

Figure 6. Spectra of rice husk and sub products procedure III.

XRD

The crystalline structures of rice husk and sub-products obtained after procedure II were analyzed by X-ray diffractometry. The XRD diffractograms are shown in Figure 7.

The curves of samples S0, SII-1, SII-Or1 showed two peaks, one near to 2θ=22 °, representing the crystalline part of the materials, and the other close to 2θ=18 °, representing the amorphous one. In the case of SII-Or2 (after NaOH treatment) three peaks are present; the peak originally centered at 2θ=22 ° was separated into two peaks (at 22 ° and 20 °) indicated that cellulose was partially transformed from cellulose I to cellulose II [4,24].

The values of crystallinity index (I_c) and relative quantity of cellulose II respect to cellulose I (I_{II-I}) of raw and treated samples from procedure II are reported in Table 1.

The values of the I_{II-I} index showed that the transformation from cellulose I to cellulose II after NaOH treatment was around 51%. The I_c values for the samples SII-1 and SII-Or1 were slightly higher than that of the original rice husk, S0, as a consequence of the partial extraction of amorphous

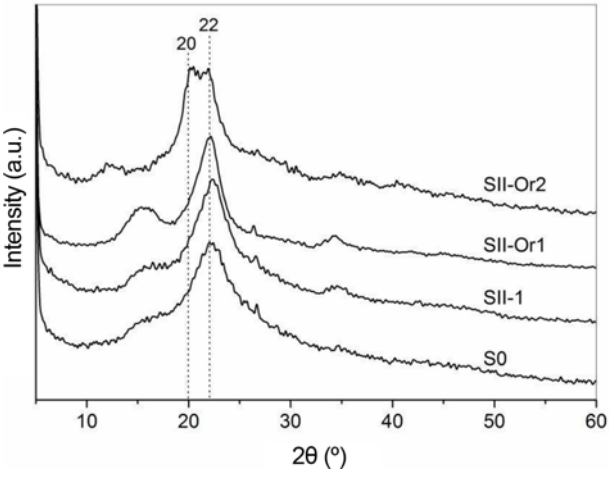

Figure 7. XRD spectra recorded from organics samples of procedure II.

Table 1. Crystallinity index (I_c) and relative quantity of cellulose II respect to cellulose I (I_{II-I}) of raw and treated samples from procedure II

Sample	I_c (%)	I_{II-I} (%)
S0	42.7	0
SII-1	46.9	0
SII-Or1	46.8	0
SII-Or2	76.7	51

components (mainly lignin and hemicelluloses). After NaOH treatment (sample SII-Or1), it can be observed an important increase in I_c (76.7 %) indicating that the last treatment completely extracted the amorphous components. The transformation from cellulose I to cellulose II was also confirmed by FTIR as it is following explained. The samples obtained before and after NaOH treatment (SII-Or1 and SII-Or2, respectively) showed a change in the characteristic cellulose bands (Figure 4) at 4000-2995 cm^{-1} (OH-stretching), 2900 cm^{-1} (CH-stretching), 1430 cm^{-1}, and 897 cm^{-1} which are especially sensitive to its crystalline structure. For the SII-Or1 sample, cellulose I, the maximum absorbance of hydrogen-bonded OH stretching was shifted from 3352 to 3450 cm^{-1} and the CH stretching band, centered at 2900 cm^{-1}, was also shifted to 2888 cm^{-1} for SII-Or2 (51 % of cellulose II) in agreement with the results previously reported by Oh *et al.* for cellulose treated with high concentrations of NaOH (between 15 and 20 % (w/v)) [25]. In addition the bands at 897 and 1430 cm^{-1}, shifted to lower wavenumber about 5 and 10 cm^{-1}, respectively, confirming the partial transformation from cellulose I to II, also observed by XRD [4,23,24].

Thermal Analysis

The TGA and DTGA allow us to study the thermal decomposition and thermal stability of rice husk and sub-products obtained after the chemical treatments. Decomposition of compounds from vegetable sources occurs in several steps in the temperature range between 200 and 700 °C [20] with the superposition of the degradation stages of the three major constituents: hemicellulose, lignin, and cellulose which decompose at dissimilar temperatures due to the differences in the chemical structure [26]. Usually, cellulose decomposition starts at 275 °C and persists until 380 °C, while hemicellulose starts its decomposition at 150 °C and continues up to 350 °C [27,28]. Finally, lignin decomposition extends to the whole temperature range, starting well below 250 °C and persisting above 550 °C [27,28].

Figures 8 and 9 show the normalized TGA and DTGA curves under air and nitrogen atmosphere, respectively, for the samples obtained after each step of procedure II. Table 2 shows the TGA and DTGA parameters for samples obtained from procedure II. Under both nitrogen and air atmospheres samples present a similar behavior. All TGA curves showed

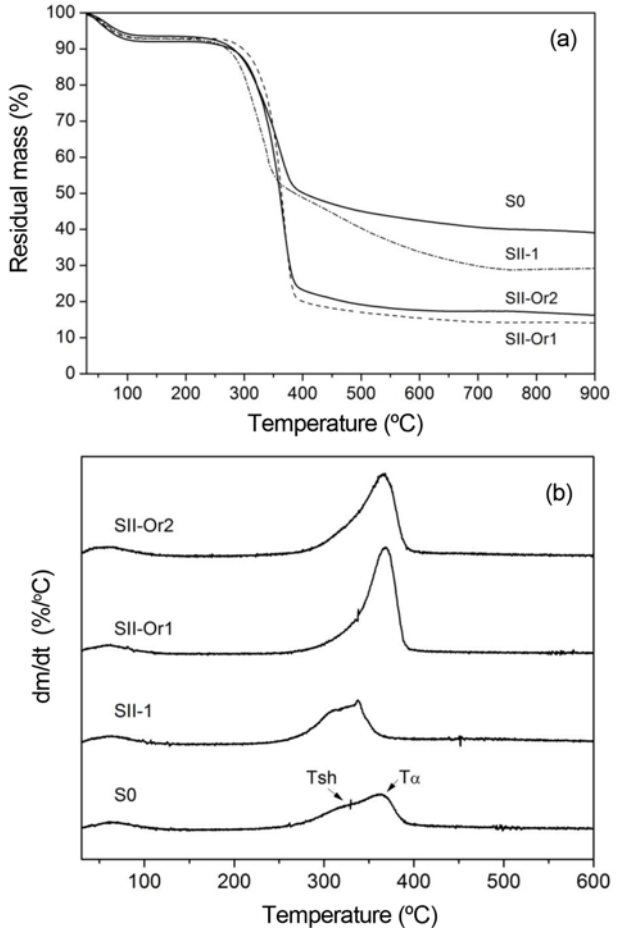

Figure 8. TGA (a) and DTG (b) curves of raw and treated samples from procedure II under nitrogen atmosphere.

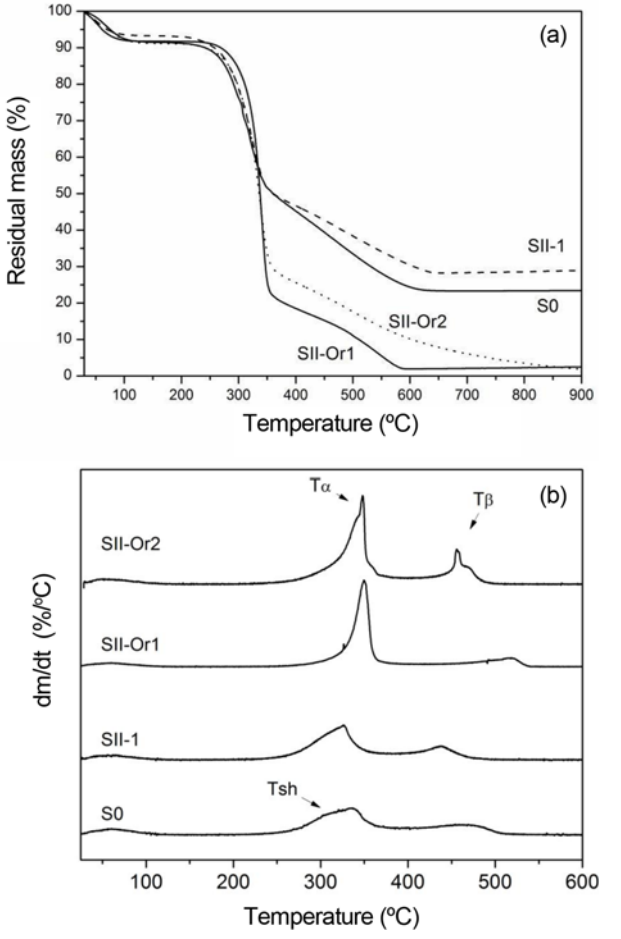

Figure 9. (a) TGA and (b) DTG curves of raw and treated samples from procedure II under air atmosphere.

Table 2. TGA and DTGA parameters for samples obtained from procedure II

Sample	Atm.	IDT (°C)	Residue (%, 750 °C)	T_{sh} (°C)	T_{α} (°C)	T_{β} (°C)
S0	N_2	288	39.0	318	362	-
SII-1	N_2	284	29.1	310	338	-
SII-Or1	N_2	310	14.3	-	368	-
SII-Or2	N_2	300	16.2	-	366	-
S0	Air	273	23.5	305	336	469
SII-1	Air	269	29.0	300	327	439
SII-Or1	Air	275	2.0	-	351	516
SII-Or2	Air	290	2.4	-	347	459

an initial weight loss around 100 °C due to water vaporization. Lignocellulosic materials absorb water at different proportions according to the content of hemicellulose, lignin, and cellulose. The moisture content was around 6.5-8.2 % for all samples.

Rice husk (S0) showed decomposition stages in agreement with the reported ranges [11,29-31]. The main peak (T_{α}) attributed to the thermal decomposition of α cellulose was around 362 °C. The shoulder at lower temperatures (T_{sh} around 318 °C) can be attributed to the thermal decomposition glycosidic links of cellulose and hemicellulose which degrade more easily than crystalline cellulose, meanwhile lignin peak is overlapped on the whole range. The sample treated with $NaClO_2$/$NaHSO_3$ (SII-1) showed a lower thermal stability which was quantified by the IDT and T_{α} values. This result is as a consequence of lignin removal leading to a higher proportion of hemicellulose in the material. After the combined treatment with KOH/HCl (SII-Or1), the silica and part of the hemicellulose were eliminated and the thermal stability increased, even compared with that of the rice husk (S0). After alkali treatment (SII-Or2), the thermal stability decreased once again, as an effect of the conversion from cellulose I to cellulose II, which was also reported in FTIR and XRD analysis.

Table 2 shows that the residual mass at 750 °C for each sample was from 14 to 39 % under nitrogen atmosphere and from 2 to 23.5 % under air atmosphere for the different samples. In the case of the treated ones, residual mass was lower as a consequence of the progressive removal of lignin, hemicelluloses, and silica. Under air atmosphere residual mass of SII-Or1 is major compared with the residue of the original sample (S0), due to the removal of organic material, lignin in this case, the percentage of inorganic material exceeds that in S0, increasing the residual mass. The weight fraction of material that still remains after heating above 700 °C corresponds to ash, that in most cases arises from the carbon content of the samples [3,20]. In the case of nitrogen atmosphere, higher residual mass at 700 °C (39 %) was observed, which is attributed to the high content of silica and carbonaceous residues of the raw material. Under air atmosphere, the residual mass was reduced in all cases because of the oxidation process of carbonaceous residues, which is in agreement with previously reported results [11,20].

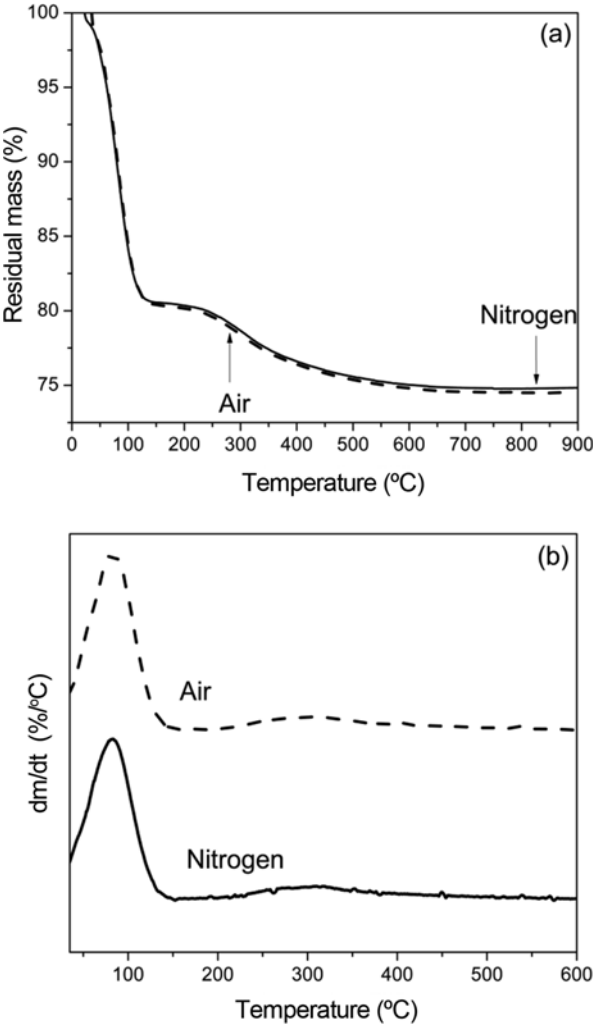

Figure 10. TGA (a) and DTGA (b) curves of the inorganic sample from procedure II before calcination (SII-In2) under nitrogen and air atmosphere.

Figure 10 displays the TGA (a) and DTGA (b) curves of the inorganic sample from procedure II before calcination (SII-In2).

This sample showed a similar behavior in both air and nitrogen atmosphere, with a maximum loss weight at 100 °C because of the hygroscopic character of silica [32]. Another peak appears around 300 °C, which is attributed to the presence of dragged organic material in the liquor rich in silica, mostly cellulose as was confirmed by FTIR.

SEM

Chemicals treatments used for the purification of cellulose induce morphological changes on the rice husk. The structure

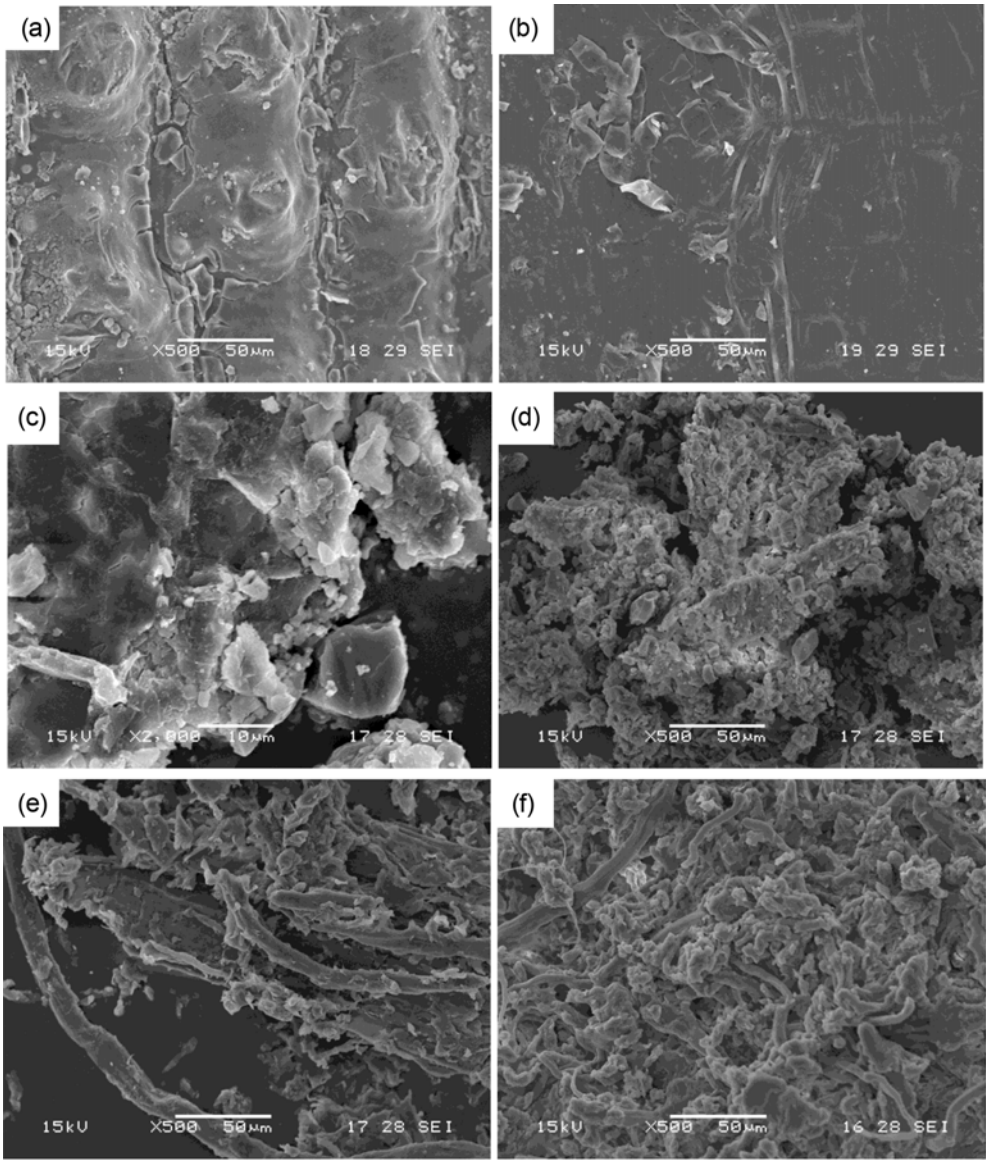

Figure 11. Morphology of original rice husk outer (a), inner surface (b), S0 (c), SII-1 (d), SII-Or1 (e), and SII-Or2 (f).

of the different samples was investigated using SEM (Figure 11).

No treated sample (Figure 11(a) and 11(b)) presented an external epidermal cell with prominent domes with a high content of silica that provides strength and stiffness to the husk and a smooth internal cell with a minor content of silica [29,31,33].

With the subsequent treatment the smooth surface of the initial sample (Figure 11(c)) becomes rougher, that fact suggested the partial removal of the non-cellulosic layer in this case of lignin confirming the results of FTIR tests (Figure 4). Removal of hemicellulose and silica in a further step enable the fibers to separate into an individual form due to the removal of all the components (mainly hemicelluloses)

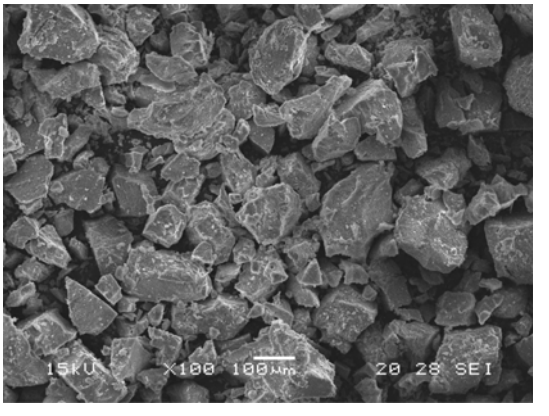

Figure 12. SEM image of silica (SII-In3).

that bind the fibers (Figure 11(e) and 11(f)).

Inorganic sample was evaluated by SEM (Figure 12). It can be observed silica particles with a broad size distribution (between 30 to 200 μm) as were expected, confirming the result of the FTIR test (Figure 5). A heating above the temperature at which it was burned would caused a formation of cristobalite and trydimite [33].

Conclusion

In this work three different procedures to extract cellulose and silica from rice husk were evaluated. The original samples and the products from each treatment were initially characterized by FTIR, showing that the procedure II was the most effective to obtain both by-products. Samples from the selected procedure were analyzed by means of TGA, SEM, and XRD to study the effect of the each step on the chemical composition and morphology. It was demonstrated that the isolated cellulose is a mixture of cellulose type I and II and that the liquor rich in silica contains small quantities of cellulose that could be eliminated by calcination at lower temperature than the usual for this process. The obtained by-products could be used as reinforcement or filler in a variety of polymeric composite systems.

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