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Performance Assessment of Deashed and Dewaxed Rice Straw on Improving the Quality of RS-based composites

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Abstract

In order to get the high performance rice straw-based composites, the synergistic effect of de-ashing or dewaxing, on the bonding behavior of rice straw (RS) fibers by eco-urea formaldehyde (UF) was assessed. The deashing and dewaxing were provided by mechanical, alkali, physical and biological treatments. The eco-UF adhesive was provided from using commercial formaldehyde-scavenger system. The chemical constituents, Fourier transform spectroscopy (FTIR) and non-isothermal thermogravimetric (TGA) analyses of un- and treated RS were estimated. The benefit of these treatments was evaluated from comparing the strength and water resistance properties of the produced agro-composites with those obtained from untreated RS bonded with UF-free scavenger, or by polymeric diphenylmethane diisocyanate (pMDI). The data showed that the extent of increasing the mean hydrogen bond strength (MHBS) value is in good relation with the efficiency of removing the silica more than wax. It is interest to note that, the modulus of rupture(MOR), and internal bond strength (IB) of board produced from relatively higher de-ashed and de-waxed RS (via mechanical followed by alkali treatment; RS-MT-AT fibers) and UF-containing scavenger are nearly the same as those values of board produced from traditional pMDI resin bonded RS of particle (~ 16.9 MPa, and 0.32 MPa, respectively). However, the thickness swelling (TS) of the investigated board is higher (17.4%), than the traditional ones (~12%). As can be seen that, biological treatment approach (RS-RS), provided higher de-waxed fibers and higher water resistance property (~13%). Among the board types tested, MOR and IB properties of RS-MT-AT and RS-BT boards meet the ANSI standard A208.1-2009 requirements for wood particleboard of class M-2. While, RS fibers provided from physical treatment (RS-PT), resulted board meet the requirements of MOR & IB of class M-S.

Keywords: Rice straw, De-ashing treatments, De-waxing treatments, Eco-urea formaldehyde adhesive, Environmentally friendly composites, RS-based composites.

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1. Introduction

In Egypt, The forest areas are about 72 000 hectares (ha) which represent about 0.1% of the land is forested (according to Food and Agriculture Organization of the United Nations [1]. This considered very small percent to cover the application of wood industry, especially with increasing the population, as well as environmentalists pressure for natural resource management and conservation have prompted the use of several woody and non-woody materials (Bagasse, rice straw

and cotton stalks) as alternative raw materials for lignocellulosic composites (particleboard, fiberboard and plywood) production.

Formaldehyde-based adhesives, especially urea-formaldehyde adhesives are used worldwide in manufacture lignocellulosic composites intended for interior applications. The drawback of this adhesive, during its curing in the hot press, some of the free formaldehyde reacts with various chemical constituents of the lignocelluloses, some is incorporated into the adhesive polymers, some is off-gassed into the air, and some is destroyed via the Cannizzaro reaction (a chemical reaction that converts formaldehyde into methyl alcohol and formic acid). Moreover, during storage or in-service use of lignocellulosic composites, especially when exposed to elevated humidity or temperatures, measurable levels of free formaldehyde can also be generated due to hydrolysis, isomerization and decomposition of formaldehyde-resin [2].

For upgrading the rice straw as a lignocellulosic material for composites produced, Boquillon et al. [3], found that the properties of wheat straw particleboards using UF resins were poor, especially for IB strength and thickness swelling. Attempts by literature investigators to improve the quality of RS-based composites were carried our through RS particles bonded with polymeric diphenylmethane dissocyanate (pMDI) resin or a combination of urea formaldehyde (UF) and pMDI [4, 5]. Combination of modified soy protein isolate with 10% of pMDI produced low density straw-protein particleboard [6]. As well as, the bond-ability of UF-bonded reed and wheat straw boards was improved by silane coupling agent and ethanol-benzene extraction treatment [7, 8]. On the other hand, most of literature focused on providing pre-treatment on wheat straw, reed, saline jose tall wheatgrass, or rice straw fibers before subjecting to board formation, e.g., alkali, enzyme, and steam pretreatment [9, 10]. The other approaches depend on producing sound absorbing composite from rice straw substitute up to 20% wood [11], and overlaying fiberboard produced from bamboo and rice straw [12]. Saadatnia et al. [13] found that composite boards made with rice straw and waste tire particles had better flexural properties than wood particleboard, insulation board, fiberboard, plywood and various other construction materials.

The foregoing literature studies were carried out on pretreatments of different straws, therefore it has not been able or lake to recommend the treatment, which affects the reduction of ash and/or wax is the most successful in motivate the production of RS-based composites, using commercial UF.

Our previous work [14, 15] in this subject showed that the properties of straw-UF and RS-pMDI particleboards are highly dependent upon the straw particle size. They also reported that steam and short durations of oxalic acid (OA) treatment resulted in significantly improved mechanical properties and dimensional stability of rice straw particleboards, especially for IB strength. Steam-treated panels exhibited slightly higher performance compared with OA-treated panels. For Studying the possibility of preparing high performance agro-based composites from rice straw by using eco-polyalcohol polymers-based adhesive system, as HCHO-free adhesive was achieved also by the

authors of this article [16]. This adhesive system provided improvement in board performance over a previously commercially HCHO-based adhesive (UF). As well as, the MOR of the produced RS-based composite exceeded the standard requirements; while IB nearly the same.

The same authors reported an improvement in the environmental performance of UF and consequently the produced sugar-cane bagasse composites when organic and inorganic materials, nitrogen-containing starch derivatives, as HCHO-scavenger [17, 18], were added. An even more effective derivative was the addition of an acrylamide-containing starch derivative. They reported also that, deashing of rice straw had a significant effect on performance of the produced active carbon [19], as well as RS is efficient agro-fibers for production cemented fiber-boards [20].

This present work deals to assess some de-ashing (as indication to de-silication) and de-waxing treatments of rice straw on the performance improvement of the produced RS-UF-based composites. The utilization of rice straw (undesirable bio-wastes) for the production of high quality bio-composite products, will add economic value, helps reducing the environmental impact of waste disposal and most importantly provide a potentially inexpensive alternative to the existing commercial artificial wood-panels.

2. Experimental

2.1. Treating of RS and characterization of fibers produced:

In this study we'll be highlight the most efficient treatments of RS fibers, which preserving to some extent its chemical constituents, besides removing the undesirable constituents (e.g., silica and wax). The mechanical and/or chemical, physical and biological treatments were carried out as follow:

- RS treated mechanically (RS-MT). RS grinded and screened through sieves with different mesh sizes (25.4mm (1 in), 19.05 mm (3/4 in), 12.70 mm (1/2 in), 6.35mm (1/4 in), 3.18 mm (1/8 in) and 1.59 mm (1/16 in) (Li, 2010).
- RS treated chemically: RS treated with a 12 % NaOH solution at 55-60 ^{o}C for 1 hour, then washed with distilled water till neutrality is reached, and dried overnight at 105°C. This material was labelled RS-AT. O**r** by treating the RS with a 3% H_2O_2 in presence of NaOH, as activator, for 30 minutes and at room temperature (~ 23 ^{o}C); (RS-HPT) [21, 22].
- RS treated mechanically followed by pulping with a 12 % NaOH solution as described in (d). This material was labelled RS-MT-AT.
- RS treated biological: by lipase enzyme (rate of activity: 25 units/mg), using water as carrier, at pH 4.8 [23]. This material was labelled RS-BT.
- RS treated physical by steaming it for 30 minutes (RS-PT).

2.2. Characterization of un-and treated RS fibers

The proximate analyses based on standard methods were used to assess the efficiency of reported treatments on structural chemistry of the RS fibrous produced. Silica based on ash content was determined by weighing samples before and after all the organic matter was burnt by heating in a muffle furnace up to 800 ^{o}C, in air, and holding this temperature for 45 minutes. Extraction of treated samples by Methanol-benzene was carried out as indication to determine the efficiency of investigated treatment on removing wax.

The contents of α–cellulose, pentosans, and lignin were also quantified according to Standard methods [24-26].

The changes in structure of treated RS fibers were also estimated from IR-spectra measurements (Mean hydrogen bond strength, and crystallinity index). IR-spectra (4000-400 cm^{-1}) were recorded on Nexus 670 FTIR spectrophotometer (Iclet Co., USA), using KBr disc. The technique of O'Connor et al. [27] was used to calculate the crystallinity index (Cr.I). The mean strength of hydrogen bonds (MHBS) was calculated according to Levdik et al., [28]. The relative absorbance of bands was calculated as the ratio of the band intensity at the subsequent wave-number to the band intensity of the wave-number at ~1320cm^{-1} that corresponds to the CH rocking of the ring.

2.3. Thermogravimetric studies:

Non-isothermal thermogravimetric analyses were performed in a Perkin Elmer Thermogravimetric Analyzer TGA7. The samples were heated in pure nitrogen (flow rate 50 mL/min) at 10 °C /minute, and within the typical temperature range: 35 – 600 ^{o}C, i.e., till no additional weight loss was observed. Measurements were made using calcined alumina as reference material. Differential thermogravimetric (DTG) peaks were examined for evidencing different behaviours between the samples, and also for understanding how the pre-treatments affected the thermal stability of fibers produced The kinetic parameters based on the weight loss data of TG curve analysis were determined according to the equations described elsewhere [28, 29].

2.4. Board manufacturing and tests

Rice straw boards were prepared by well blending the sprayed adhesive with particles of un- and treated rice straw particles. An adhesive content of 16% for UF resin was used based on the weight of oven-dried rice straw particles. 4% for pMDI resin, as the traditional adhesive for silica-containing fibers, was also used for comparison [14]. To provide eco-friendly UF adhesive and based on our previous work dealing the reduction its free-HCHO to about ~73 % [17]; .amide-containing starch derivative was added together with urea formaldehyde and 0.5% paraffin wax. A hot press was used to manufacture the boards and the platen temperature was fixed at 170 ^{o}C for the manufacturing of UF-bonded boards and 180 ^{o}C for the manufacturing of pMDI-bonded boards. In the case of UF

and pMDI resin-based boards, the total hot press time was 510s and 350s, respectively. Two replicate boards were made at each condition.

For mechanical tests: Modulus of rupture (MOR) and internal bond (IB) values were tested using Instron Universal Machine. The boards were tested and evaluated in accordance with American standard test methods for evaluating properties of wood base fiber and particle panel material [31]. Each measurement presented herein is the average for six samples cut from two different boards.

For water resistance property: thickness swelling (TS) was measured following the standard set forth in ASTM D 1037-06a [31]. The TS was measured after 24-h immersion in distilled water at 20^{o}C. Six replicates were made to evaluate the dimensional stability of boards for each condition.

2. Results and Discussion

3.1. Characterization of rice straw fibers

The ash content and the MeOH-Benzene extractive percentages of treated rice straw are shown in Fig. 1. These analyses were carried out as indication to the efficiencies of these selected treatments (mechanical, chemical, biological, and physical), for removing silica and wax. It is interest to find out what is the effect of screening, as easy way, on reducing the ash content by about 34- 42 %, at particle size 3.18-6.35 mm. This is ascribed to the un-split hollow stems exist in the particle mat when the dimension of the opening size was greater than 6.35 mm. Therefore, mesh size 6.35 mm was used for further treatment. For the effect of further treatment, it is clear that, the best effect, especially for reduction of ash, is obtained by treating RS with mechanical followed by alkali (RS-MT-AT), in comparison with alkali treatment (RS-AT). Where, the reduction in ash and wax contents is increased from 52.7 % to 55.5% and from 78.58% to78.72%, respectively.

Biological and physical treatments are significant effect on reducing wax content than ash content of RS. Whereas, the extract content of RS-BT with MeOH-benzene decreases of 63.5%; while the ash content decreases of 32 -35.5%. Treatment by H_2O_2 is slightly affect on both silica and wax (reduction, % are 14.45 & 22.5, respectively).

The data from using alkali, biological, physical and/or mechanical treatments should have a positive effect on adhesion of a resin on RS substrate. Based on these noticed, the RS-fibers from these efficient treatments were subjected for further study the effect of these treatments on chemical constituents, IR-spectra and thermal analyses of RS.

Figs. 1 and 2 show that, the mechanical treatment of RS provided a decrease in α-cellulose of raw RS from 54.6 to 47.2 %,.This is related to such treatment given that RS-MT particles bigger than 3.18 mm; while smaller particles which were eliminated by sieving contain much more ashes. The removal of the latter therefore produced the increase of pentosans and klason lignin contents in RS-MT. As expected, given the acidic character of silica, alkaline treatment by NaOH is the most efficient one for reducing the ash content and extract, only 8.1 % and 1.9% are remained in RS-AT.

Such a pre-treatment also produced the increase of both α-cellulose and klason lignin contents, as compared with raw RS, whereas the amount of pentosans drastically decreased. Mechanical treatment followed by alkaline treatment produced a material (RS-MT-AT) with higher reduction in ash content (~ 56%) and preserving the content of α-cellulose (~54.3%), which is closed to that content of the original RS (54.5%). However, the contents of pentosans and Klason-lignin are found to be increased by the investigated treatment (15 % & 24%), respectively, due to the combined favourable effects of MT and AT.

Biological treatment by lipase enzyme (RS-BT) is also significant affect on decreasing the wax and pentosans contents by about 63.5% and 31%, respectively; while led to increase the α-cellulose and klason-lignin contents (56.2% & 20.6%, respectively), For the case of treated the RS by steam (RS-PT), this led to produced fibers with relatively higher klason contents and pentosans (21.9 % & 18.8%, respectively), without significant reduction in α-cellulose content (~53.2%), as well as provided relatively higher degree of de-waxed, whereas the reduction in MeOH-Benzene extract reached to ~ 64%..

For IR-spectra study, this analysis was employed to selectively follow the chemical functionality changes of RS fibers on treatment by the foregoing treating agents. The IR-spectrum of RS (Fig. 2 and Table 1), shows characteristic bands at 3433 cm^{-1}, 2925 cm^{-1} due to OH , CH (stretching vibration) and hydrogen bonds (intra & inter molecular hydrogen bonds). The bands at 1727 cm^{-1}, 1637 cm^{-1}, corresponding to C=O (stretching vibration); while the bands at 1441.5 cm^{-1}, 1373 cm^{-1} and 899.6 cm^{-1} corresponding to OH (bending vib.), OH (phenolic group of lignin and CH (rocking vib.), respectively. The ratios of absorbencies at 3433 cm^{-1} to that at 2900 cm^{-1} (indicates to MHBS), and at 1440cm^{-1} to that at ~900 cm^{-1} (indicates to Cr. I), of RS fibers before and after treatment are listed in Table 2-b. It can be shown that, most of foregoing treatments are found to favor the formation of hydrogen bonds, and red shifting the band maxima corresponding to stretching vibration of OH (from 3433.6m c^{-1} to 3406-3421 cm^{-1}), with the increase in its absorbance intensity. This is probable ascribed to removal of undesirable components (silica and wax), which leads to enhance the intra molecular hydrogen bonds between fiber chains. The decrease in the band absorption related to Si –O and O-Si-O stretching at the region of 1300-1000 cm^{-1} [32], confirms this view.

The extent of increasing MHBS values (Table 2), is related to the efficiency of removing the silica more than wax, as manifested from exciting a good relation between increasing the MHBS with the efficiency of removing the silica-based ash and wax contents, by chemical treatments, as clear in the following relations:;

MHBS= 0.041 + 0.036 (% Silica removal) …….. (r= 0.9143, SD= 0.2306)

MHBS= 0.2115 + 0.0513 (% Wax removal) …….. (r= 0.964, SD= 0.1514)

While, there is no good relation linked the efficiency of removing the undesirable constituents (silica & wax) in RS fibrous and crystallinity indices, with changing the treating agent. (as clear from the r & SD values of the following equations).

Cr. I.= 2.7 + 0.045 (%Silica removal)……..(r= 0.675, SD= 1.093)

Cr.I = 3.03 + 0.023 (% Wax removal %)……(r= 0.47025, SD=1.463)

This means that the crystallinity index of treated RS fibers is depended not only on removing efficiency of silica and wax surrounding the fibers, but also on efficiency of removing the low molecular weight hemicelluloses (amorphous holocellulose). Whereas, the removal efficiency of pentosans in case of RS-AT (~ 59%), is higher than the other treated RS fibers. This trend is however not observed for biological treatment by lipase enzyme, RS-BT, where the resulted fibers had lower pentosans & lower Cr.I. It means that the biological treatments not led to attack and eliminate the hemicelluloses but let to degrade the linkage between cellulose and lignin. This view is emphasized from lower decreasing the relative absorbance related to phenolic OH and ether linkage, compared to other treated fibers.

For thermal stability study**,** the TGA and DTG curves of untreated RS fibers (Fig. 3-a & Table 3), show three degradation stages. At lower temperature, i.e. < 112^{o}C (1st degradation stage), the weight loss is due to the evolution of sorbet moisture. The second process, in the range from166-328 ^{o}C is due to the decomposition of RS components, leading to the formation of carbonaceous char. This is followed by weight loss within the temperature of 328 -484 ^{o}C due to oxidation of charred product. The 2nd and 3rd process regard the main degradation stages.

For treated RS (Figs. 3-a & 3-b, Table 3), the start temperatures (onset Temperature) of the 2nd degradation stages are higher than untreated RS. This is due to the removal of wax and silica beside the low molecular weight hemicellulose (amorphous holocellulose). As can be noticed that, maximum raise in the start temp. is noticed for alkali treated RS fibers (RS-AT), however the lower onset temperature was noticed for biological treated RS fibers This is ascribed to the formation of carboxylic groups ($E_{C=O}$ is relatively higher and equal to 1.47; Table 1), beside the removal of silica and wax in the former treated fibers. This led to enhance the formation of intra molecular hydrogen bonding with hydroxyl groups, in other glucopyranose units. Therefore, more energy is needed for decomposition (Table 3). This view confirms the increase in Cr. I of alkali treated RS, than other treatments. While, in the later treated fibers (RS-BT), the ether linkage of lignin with hollocellulose is cleaved, as manifested from relative absorbencies related to OH-phenolic and ether linkage at 1349 cm^{-1} and 1120 cm^{-1}, respectively (Table 1).

Table 3 also shows that, thermal stability of the foregoing treated samples increase in the order:

RS-AT > RS-MT > RS-MT -AT > RS-PT > RS-BT.

3.2. Effect of degree of deashing and dewaxing by treatments on properties of RS-based composites.

The experimental results of foregoing treated RS fibers, and bonded with 16% UF-amide-containing starch adhesive system resin, in presence of 0.5% hardener and 0.5% paraffin wax emulsion, are shown in Fig. 4, in comparison with that obtained from using 4% pMDI adhesive.

By changing the chemical and biological processes of de-ashing and de-waxing of RS fibers, the improvements in board properties have occurred, compared to board made from screened RS fibers, with particle size 6.35 mm.

For the improvement in modulus of rupture (MOR) and internal bond (IB), it is observed that using UF-AM-cont. Starch adhesive system provided greater improvement than UF adhesive. For the case of using UF adhesive, the best bonding results are obtained by mechanically pretreatment RS fibers, followed by alkali (RS-MT-AT). While on using UF-AM-containing starch adhesive system, both RS-MT-AT and RS-BT, as substrates, provide boards with best mechanical properties. This indicates that the alkali and enzyme treatments are effective in removing waxes together with silica This should have a positive effect on adhesion of a UF-adhesive system on such substrates.

While, for thickness swelling property it was noticed that, board made from RS-BT had relatively low thickness swelling than those produced from other pretreated fibers.

It is interest to note that MOR & IB of board produced from RS-MT-AT fibers and UF are nearly the same as those values of board produced from MDI resin bonded RS of particle size 6.35 mm (~ 16.9 MPa, and 0.32 MPa). While, the thickness swelling of the former board is higher (17.4%) than the latter ones (~12%).

On comparing these obtained results with the literature reported data, it was recommended that, mechanical pretreatment followed by alkali treatment of RS (RS-MT-AT), and using UF-scavenger adhesive system motivate such undesirable waste for providing particle board with the superior qualities than those made with from alkali treated saline Jose Tall Wheatgrass and UF adhesive; MOR and IB were 4.4 MPa and 0.13 MPa, respectively [9]. Moreover, this board with properties nearly similar to that produced from Aspergillus niger cellulases treated wheat straw also using UF, MOR 16.3 MPa and IB 0.42 MPa [23]. Also, we recommended that, the priority of particle-board produced in this article than the literature reported boards in this safety adhesive system.

Among the board types tested, MOR and IB properties of RS-MT-AT and RS-BT boards meet the ANSI standard A208.1-2009 requirements for wood particleboard of class M-2. While, RS-PT board meet the requirements of MOR & IB of class M-S [33].

4. Conclusion

In this article an effort has been made to demonstrate the production of high quality lignocellulosic composites from rice straw as undesirable agro-waste, and commercial urea-formaldehyde adhesive. The role of de-ashing and de-waxing treatments on the performance of composites, and which one is more benefit were assessed. The preliminary characteristic of treated RS fibers by FTIR-spectra and non-isothermal TGA analysis were carried out. Our results demonstrated that the strength properties of board produced from higher de-ashed and de-waxed RS fibers) and UF-containing scavenger are nearly the same as those values of board produced from high cost pMDI resin bonded RS particles. While, higher de-waxed treatment, via biological treatment provided higher water resistance property. Among the board types tested, MOR and IB properties, the produced boards meet the ANSI standard A208.1-2009 requirements for wood particleboard of class M-2.

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Table 1. Main IR-absorption bands of un- and treated rice straw.

Particle size	ν_{OH} or ν_{NH} (stretching)	ν_{CH} (stretching)	$\nu_{C=O}$ (stretching)	ν_{OH} or ν_{NH} (bending)	ν_{-OH} (phenolic lignin)	ν_{-O-} (ether linkage)	ν_{CH} (rocking)
Rice straw	3434	2925	1727 1637.3	1426	1386	1103	896.7
RS-MT	3420	2920.7	1642 1513	1428	1370	1160	898
RS-AT	3421	2921.6	1640	1427	1376	1159	878.4
RS-MT-AT	3420	2919.7	1640	1429	1372	1161	878
RS-BT	3779 3697 3406	2921	1723.1 1599.7	1429	1349	1120	900
RS-PT	3779 3699 3409.	2921.6	1722.2 1602.6	1412.7	1370	1102	912.7

Table 2. IR-measurements of un- and treated rice straw

Sample	MHBS ($A_{OH(Str.)}$ /$A_{CH\ (Str.)}$)	Cr.I. ($A_{\sim1430\ cm^{-1}}$ /$A_{\sim900\ cm^{-1}}$)
Rice straw	1.369	3.606
RS-MT	2.172	3.812
RS-AT	1.993	5.490
RS-MT-AT	1.854	6.314
RS-BT	1.013	2.874
RS-PT	1.280	3.552

Table 3. Kinetic parameters of untreated and treated rice straw

Sample code in chart	stage	Temp. range °C	DTG peak temp,. °C	"n"	E_a kJ/ mole	Wt. remain, %
RS	1st	50- 112	63	-	-	95.86
	2nd	166-328	254	1.0	79.37	45.85
	3rd	328-484	440	1.0	91.95	22.59
RS-MT	1st	50 - 99	70	-	-	91.35
	2nd	190- 313	262	1.5	132.79	45.36
	3rd	350- 495	414	1.0	130.11	19.80
RS-AT	1st	50- 109	67	-	-	90.78
	2nd	210 -324	298	0.5	111.83	37.45
	3rd	324 - 465	407	1.0	111.80	9.13
RS-MT-AT	1st	50- 92	59	-	-	92.84
	2nd	181 -328	275	3.0	100.77	40.24
	3rd	328- 471	411	2.0	142.28	15.78
RS-BT	1st	50- 85.5	55	-	-	93.29
	2nd	177 -321	292	0.5	96.31	34.52
	3rd	350- 430	290	1.0	168.79	15.93
RS-PT	1st	50- 92.3	67	-	-	94.10
	2nd	187 -324	294	0.5	99.81	36.23
	3rd	345- 450	403	1.0	194.44	21.34

Where, n:: order of degradation reaction,
Ea: Activation energy of degradation.

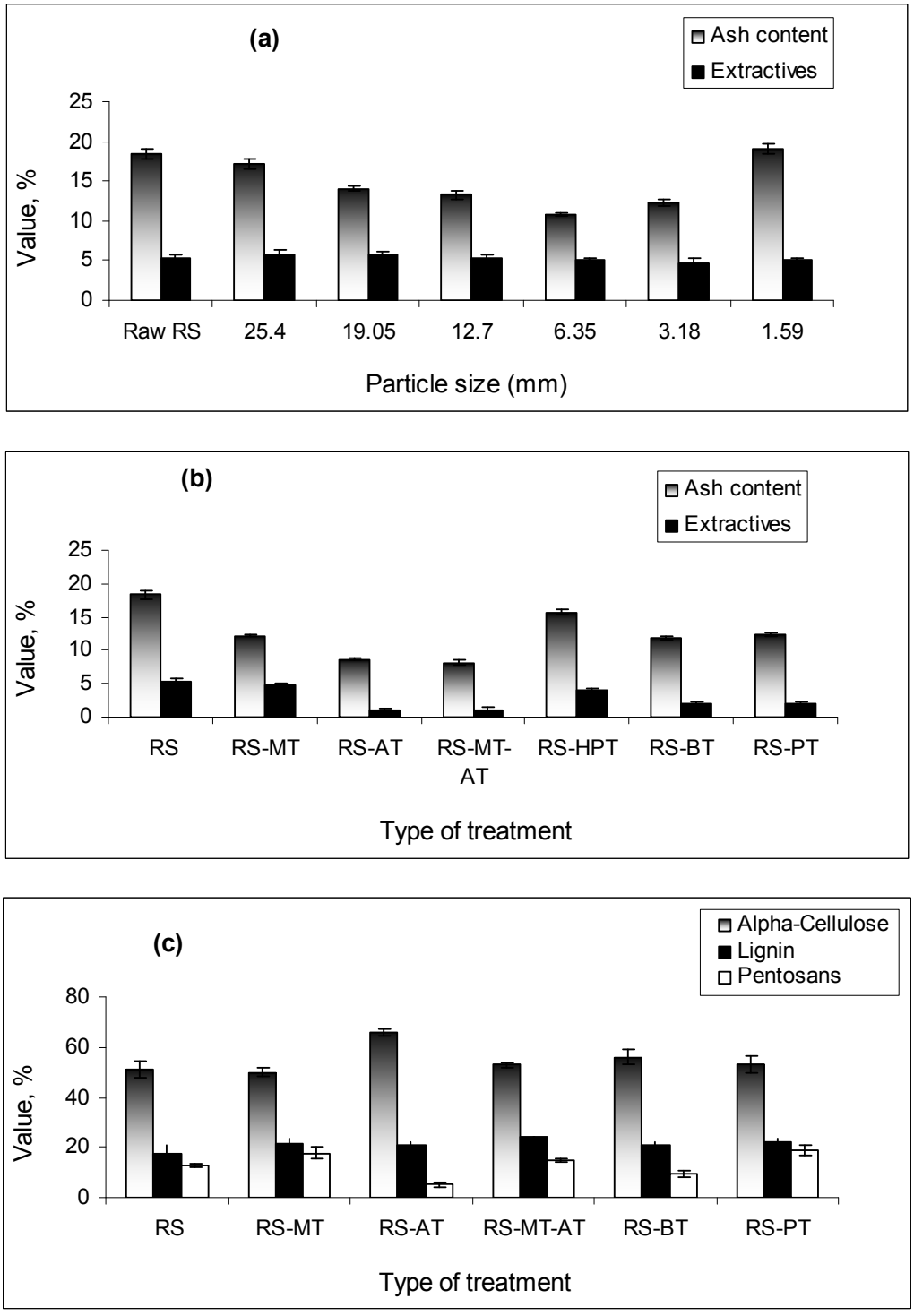

Fig. 1 Removal efficiency of silica and wax of RS as a function of treatments. (a) Mechanical Treatments, (b) Chemical treatments, (c) chemical constituents of RS fibers.

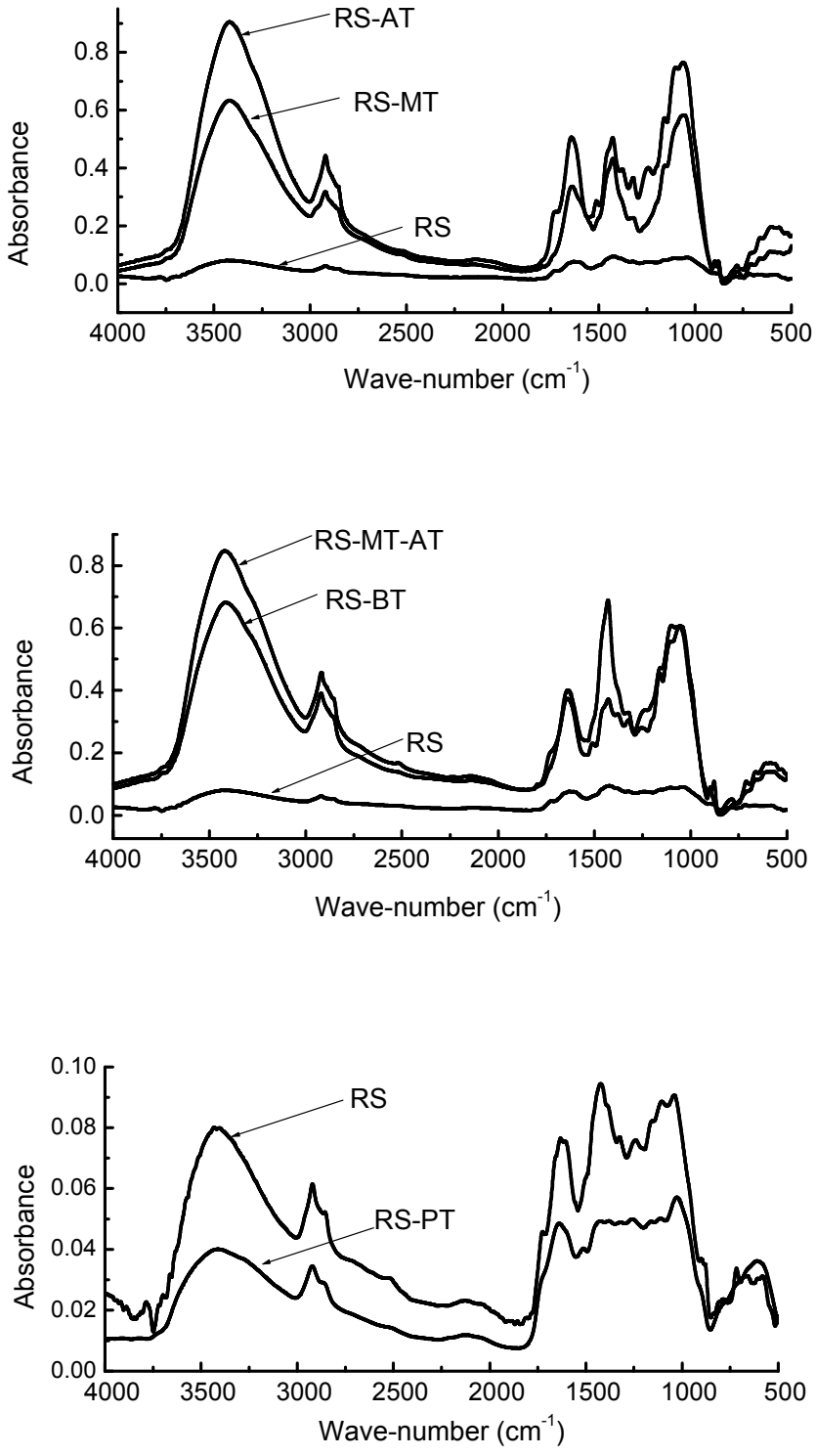

Fig. 2 FT-IR Spectra of untreated and treated RS fibers.

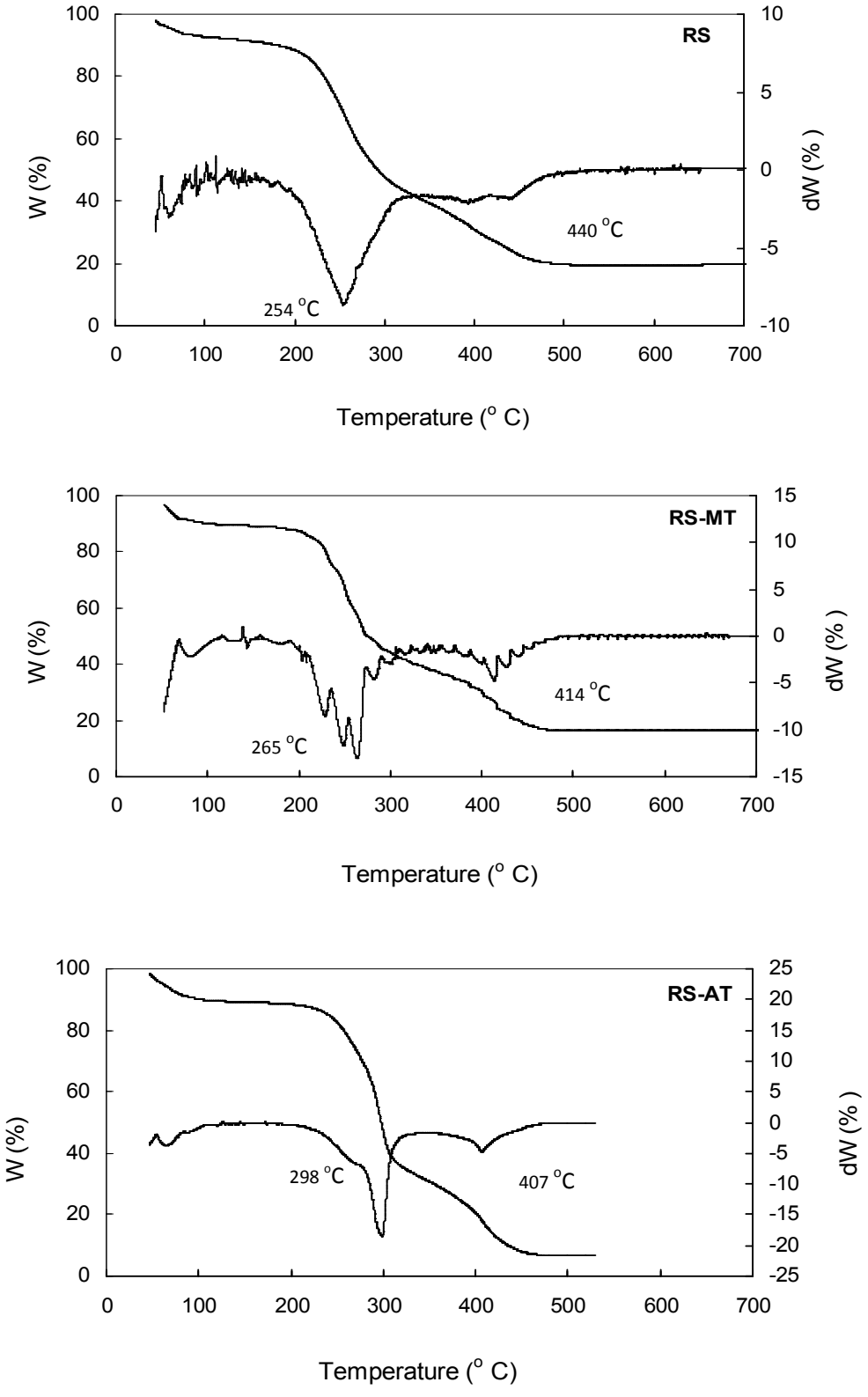

Fig. 3-a TGA and DTG curves for untreated and treated RS fibers.

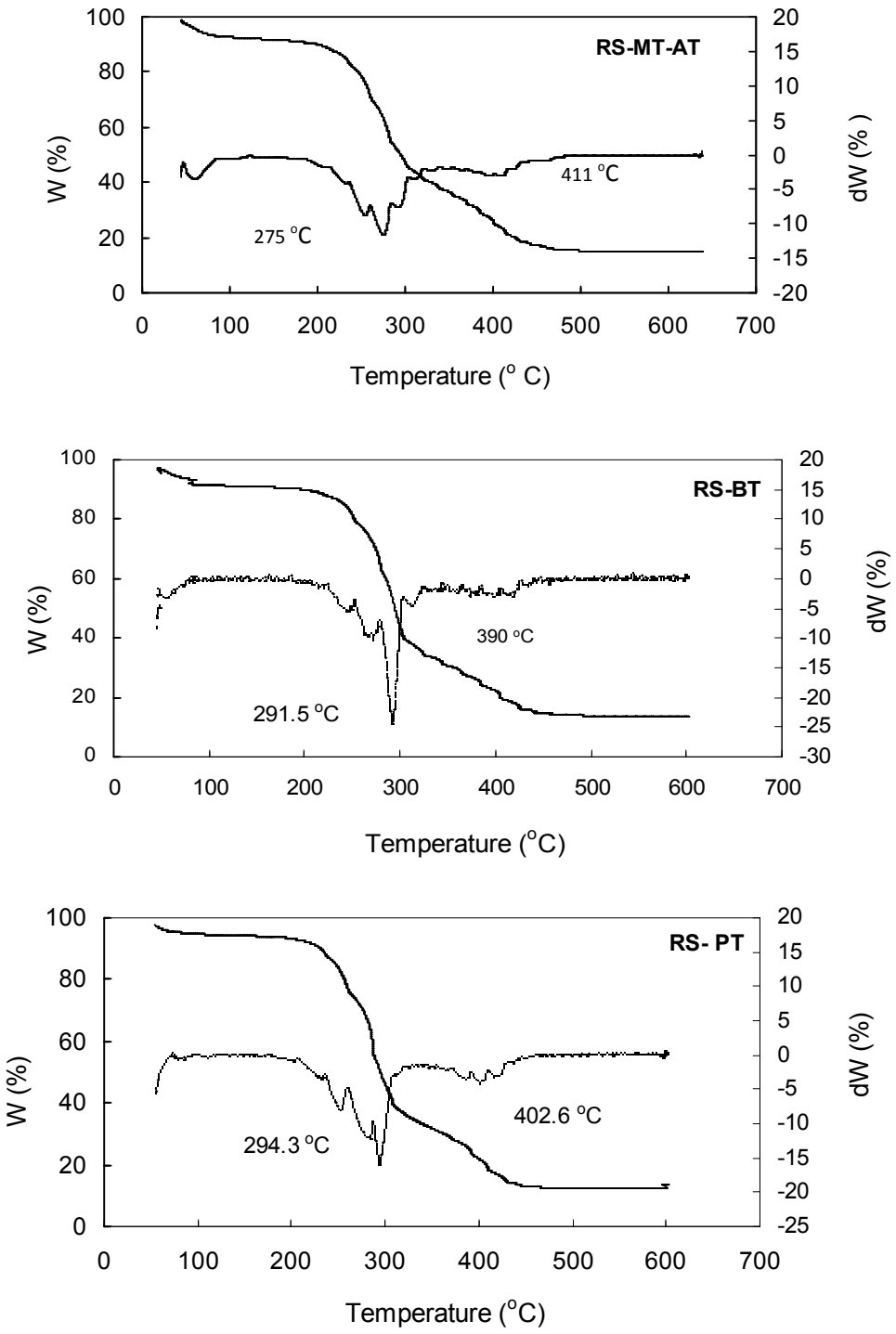

Fig. 3-b TGA and DTG curves for treated RS fibers.

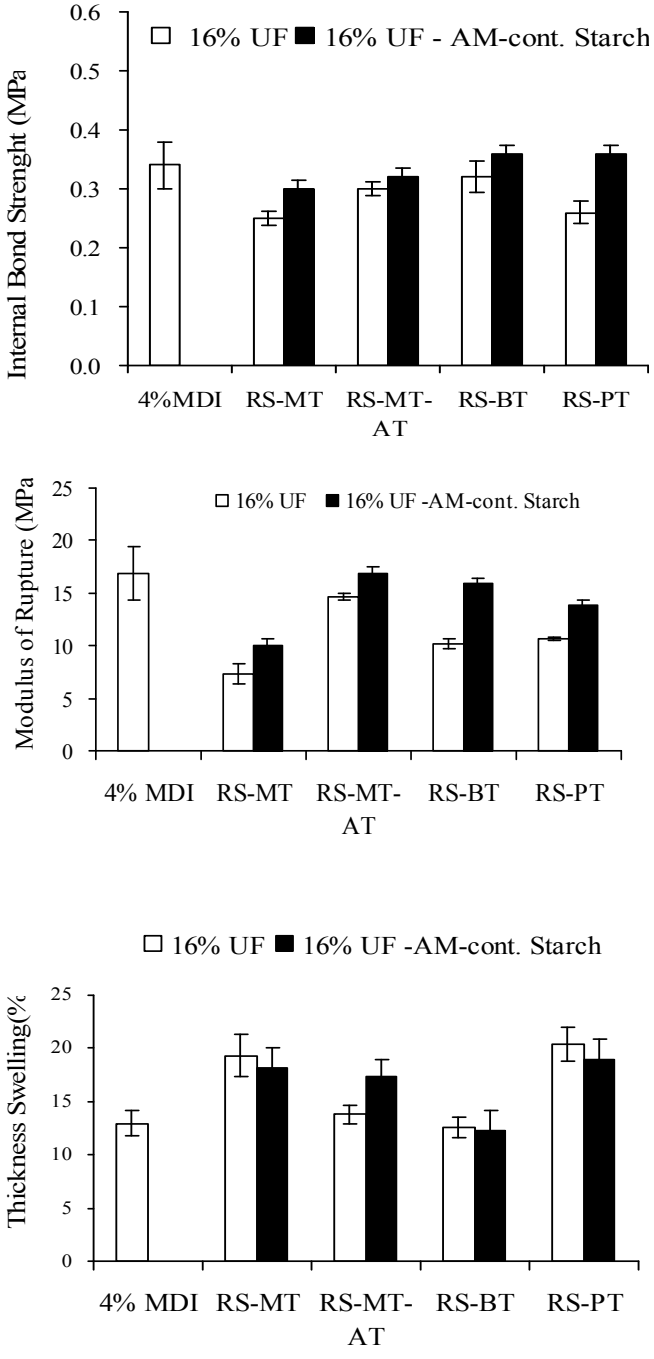

Fig. 4. Effects of treatment process of RS fibers on properties of produced rice straw –based particleboard.