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EFFECTS OF STEAM CURING AND ALKALI TREATMENT ON PROPERTIES OF RICE HUSKS

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ABSTRACT

Steam curing and alkali treatments of agricultural fibres are used to improve their adhesion properties. However, this should be achieved with minimum damage to the fibre's properties. This paper reports the effects of steam curing and alkali treatments on the properties of rice husks. Steam curing involved heating a portion of rice husks with steam between 100 and 140°C. Alkali treatment was done by soaking the other portion of rice husks in 2 to 8% NaOH solution at 27°C for 24 hr. Untreated and treated rice husks were then examined to determine their chemical contents and the effects of the treatments on their thermal stability and surface roughness. Results have indicated that chemical, physical, and thermal properties of rice husks were not significantly affected by steam curing. On alkali treatment, the inflection temperature of treated rice husks was found to decrease from 356 to 324°C, while the intermediate degradation temperature, which was observed in untreated rice husks at 218 to 334°C, was absent. The final residue after heating untreated rice husks to 700°C was 8% higher than that of alkali treated rice husks. Lignin and hemicellulose contents decrease by 96% and 74%, respectively, in alkali treated rice husks when the concentration of NaOH was higher than 4%. Surfaces of alkali treated rice husks with concentrations of NaOH greater than 6% were also considerably damaged. Thus, steam curing between 100 and 140°C causes less effect on the properties of rice husks than alkali treatment as reflected by decrease of the internal bond strength of the particleboards.

KEYWORDS: alkali, chemical contents, degradation, rice husks, steam, thermal stability, surface roughness, treatment.

INTRODUCTION

Rice husks are among few agricultural residues, which are easily obtained in large quantities at one location. They burn slowly, do not easily undergo degradation, and are very abrasive due to high silica content. The disposal nuisance of the large quantities of rice husks generated annually, and a need to exploit their high fibre contents with little energy input [1] have sparked a considerable number of studies on utilization of this resource for manufacture of composites [2,3,4]. Despite these efforts, commercialization of particleboards manufacture from rice husks has not been very successful because a substantial amount of resin is required for rice husks in order to produce particleboards of suitable properties [4,5]. The nature of physical properties and chemical composition of rice husks are responsible for this. Rice husks have low bulk density [6] and consequently require high pressures to produce boards of acceptable properties. When the chemical composition of rice husks is compared to those of other agricultural feedstocks used in the manufacture of particleboards, rice husks have high lignin content and about 20% of their outer surfaces are covered by silica in conjunction with cuticle [7,8]. So, most compatible binders are required to produce stronger interface bonds between rice husk's surface and matrix. However, due to high resilience of the rice husk particles [9], binders that are much stronger than conventional resins are required for obtaining strong interfaces that can withstand the internal stresses generated during spring-back of compressed rice husks particles [4,16]. Therefore, large amounts of expensive binders are often needed to produce stronger particleboards.

Improvement of the adhesion properties of rice husks requires modification of their physical and chemical surface properties. The most common techniques used for surface treatments of agricultural fibres and particles include steam and chemical treatments. Steam explosion is a high pressure and high temperature process, which removes impurities from the fibre surfaces such as silica, waxy, and pectin, and produces clean and rough surfaces with cellulosic microfibrils [10,12]. The purpose of this is to improve the adhesion properties of the fibre surfaces without significantly damaging their composition and properties. Chemical treatments are among the most popular methods used for the surface treatments of agricultural fibres. The most common chemical treatment method is alkali treatment using caustic soda (NaOH). This method improves the adhesion properties of agricultural fibres by facilitating the interaction between the fibre surfaces and the matrix [13,14]. Albano *et al.* [15] associated this improvement with partial elimination of lignin and hemicelluloses from the surfaces of alkali treated fibres. However, the most important issue to note during alkali treatment is whether the changes achieved do not affect the properties of the fibres. A study on production of composite panels from steam and alkali treated rice husks have revealed that the temperature of the steam and the concentration of the caustic soda have significant effects on the mechanical properties of the composite panels [16]. Thus, from this observation, a thorough interpretation of the influence of treatments on the composite properties needs also to include the study of the effects of the treatments on the fibres properties. This paper reports the results of a study carried out to determine the effects of steam curing of rice husks from 100 to 140°C and alkali treatment with 2 to 8% NaOH (w/v) on surface roughness, chemical contents, and thermal stability of rice husks. Lignin and crude fibre content of rice husks have been related to the internal bond strength of the rice husk composite panels.

EXPERIMENTAL

Materials

The main raw material in this study was rice husks. The rice husks were obtained from Morogoro Tanzania. The mould release agent for phenol formaldehyde resin was PAT 607/PCM supplied by Wurtz Trennmittel und Additive in Germany. Local chemical dealers supplied NaOH.

Treatments of Rice Husks

Steam curing of rice husks involved heating a portion of rice husks in the low-pressure steam reactor for 60 min using steam supplied from the boiler in the 100 to 140°C temperature range, being the maximum capacity of the equipment. After cooling overnight, the treated rice husks were dried at 102°C for 6 hrs in an air-circulated oven. Alkali treatment involved soaking the other portion of rice husks in 2%, 4%, 6% and 8% solutions of NaOH, respectively, conditioned at 27°C for 24 hrs. The alkali treated rice husks were then rinsed thoroughly in water to remove NaOH before they were sun-dried for about 1 to 2 days. Properties of rice husks before treatments were assumed equal. During treatments, steam or NaOH was assumed to attack equally all the rice husk particles.

Fabrication of the Particleboards

The particleboards were manufactured by spraying phenol formaldehyde (PF) resin onto the fibres in a rotating mechanical mixer. The mixture was then consolidated by a 50-ton hydraulic hot-press at 170°C for 13 min. The maximum compaction pressure was preset to 4.4 MPa.

Surface Roughness of Rice Husks

JEOL-JSM 35 CF scanning electron microscope was used to study the surface roughness of untreated and treated rice husks. The samples were sputtered with gold before scanning.

Chemical Contents of Rice Husks

Proximate analysis of chemical contents of rice husks before and after treatment was carried out by gravimetric and colorimetric techniques. The tests were replicated three times.

Thermal Stability of Rice Husks

METTLER TOLEDO TGA/SDTA 851^e Thermal Gravimetric Analyzer was used to determine the thermal stability of rice husks before and after treatment. The samples were heated from 25 to 700°C under N_2 gas at a flow rate of 55 ml/min.

RESULTS AND DISCUSSION

Effects of Treatments on Surface Roughness of Rice Husks

Figures 1 to 5 show the scanning electron micrographs of the outer surfaces of untreated rice husks (Figure 1), rice husks treated by 2%, 4%, 6% and 8% NaOH solutions, respectively (Figures 2 to 4), and husks steam cured at 130°C (Figure 5). The results show that the outer surface of untreated rice husks is quite rough. The roughness of the outer surface is represented by the asperities (Figure 1). However, after alkali treatment the asperities began wearing out gradually as the concentration of NaOH was increased from 2% to 8%. The asperities were almost completely removed from the surface after treating the rice husks in solutions of 6 to 8% NaOH (Figure 3 and 4). Effects of alkali treatments on the change of surface roughness of various agricultural fibres are widely acknowledged [14]. The surface roughness increases when impurities on the fibre surfaces including lignin and hemicelluloses are removed [12,13]. The effect can be very destructive as shown in Figure 4 where the particle became weaker and started to develop cracks after treating it with 8% NaOH. However, steam curing at 130°C did not cause any significant change of surface roughness of the outer surface of rice husks (Figure 5). The asperities observed on the outer surface of untreated rice husks in Figure 1 are similar to those of rice husks cured at 130°C. This indicates that steam curing at 130°C was not adequate to cause significant changes of the surface roughness similar to alkali treated rice husks. It is reported that significant changes of the surface properties of agricultural fibres such as defibrillation can be achieved when the fibres are treated at high pressures and high temperatures exceeding 200°C [10]. These temperatures were not reached because of the equipment's maximum capacity.

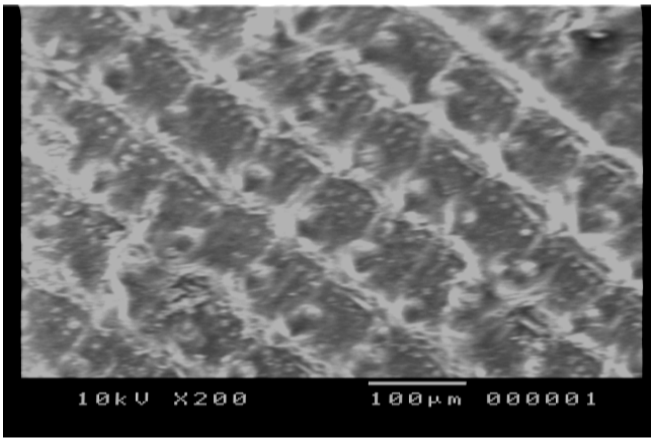

FIGURE 1- SEM MICROGRAPH OF ALKALI TREATED RICE HUSKS 2% NaOH

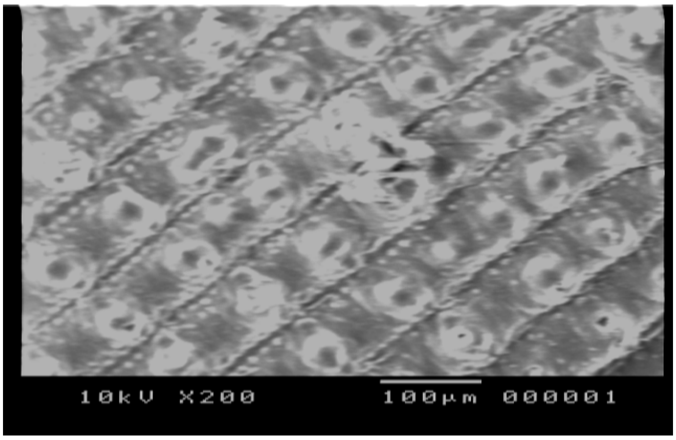

FIGURE 2- SEM MICROGRAPH OF ALKALI TREATED RICE HUSKS 4% NaOH

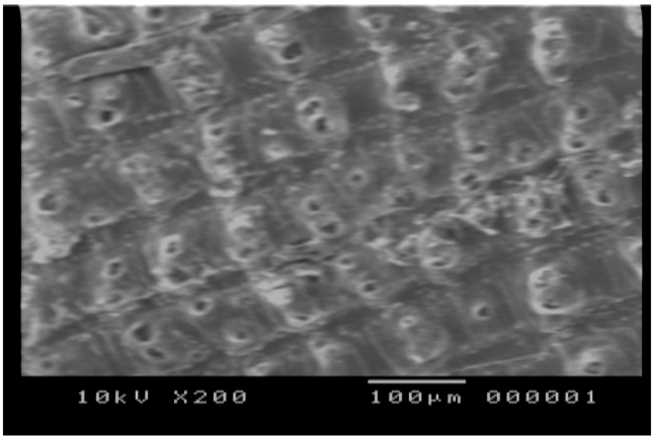

FIGURE 3- SEM MICROGRAPH OF ALKALI TREATED RICE HUSKS 6% NaOH

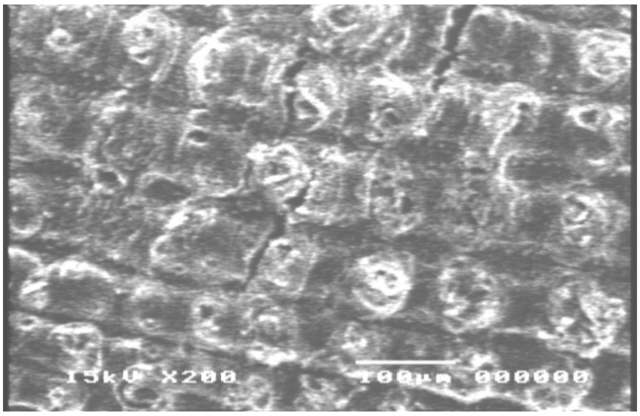

FIGURE 4- SEM MICROGRAPH OF ALKALI TREATED RICE HUSKS 8% NaOH

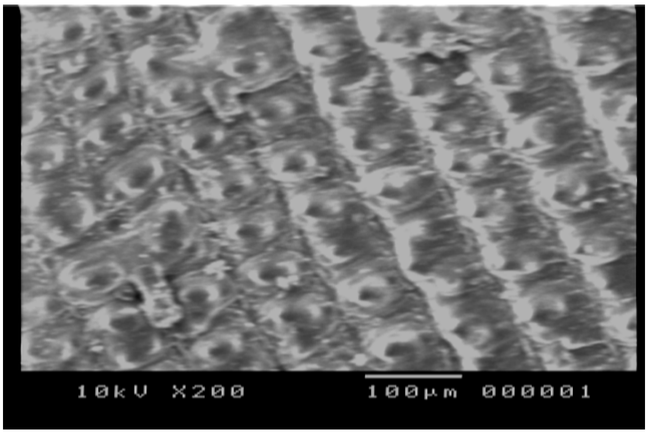

FIGURE 5- SEM MICROGRAPH OF STEAM TREATED RICE HUSKS 130°C

Effects of Treatments on Chemical Content of Rice Husks

Results of chemical composition analysis of untreated and treated rice husks are indicated in Figure 6. Alkali treatment caused substantial chemical degradation of hemicelluloses, lignin, and part of the crude fibres in rice husks as the concentrations of NaOH were increased from 4% to 8%. The composition of hemicelluloses decreased by 74% when the rice husks were treated by solutions of NaOH higher than 4%. Lignin content decrease by 96% as the concentration of NaOH was increased to the same range. Albano *et al.* [12] using 18% NaOH and Rajulu *et al.* [17] using 2% NaOH have also confirmed elimination of lignin and hemicelluloses in alkali treated agricultural fibres. However, it is also believed that lignin can retain its structural stability [18] or possibly form a lignin-cellulose structure [21] instead of degrading when lignocellulosic fibres are treated in NaOH. Supposedly, lignin can retain or change its structure to form lignin-cellulose during alkali treatment but this depends on the concentration of NaOH, treatment time, and temperature, or the method used to analyse the fibres. The results of chemical content analysis have demonstrated that lignin degraded during alkali treatment and its degradation was very substantial at higher concentrations of NaOH greater than 4%. However, the silica content in rice husks was not affected by alkali treatment at the concentrations of 2 to 8%. Steam curing of rice husks at 120°C and 140°C did not affect their chemical content. Previous work by Erins and Belickis [12] has indicated that steam treatment at higher-pressure levels and temperatures exceeding 200°C can change the chemical composition of rice husks. This implies that steam curing of rice husks between 120°C and 140°C was not adequate to change the chemical content of rice husks. This change can be achieved by treating the rice husks at higher steam temperatures or with higher concentration of NaOH.

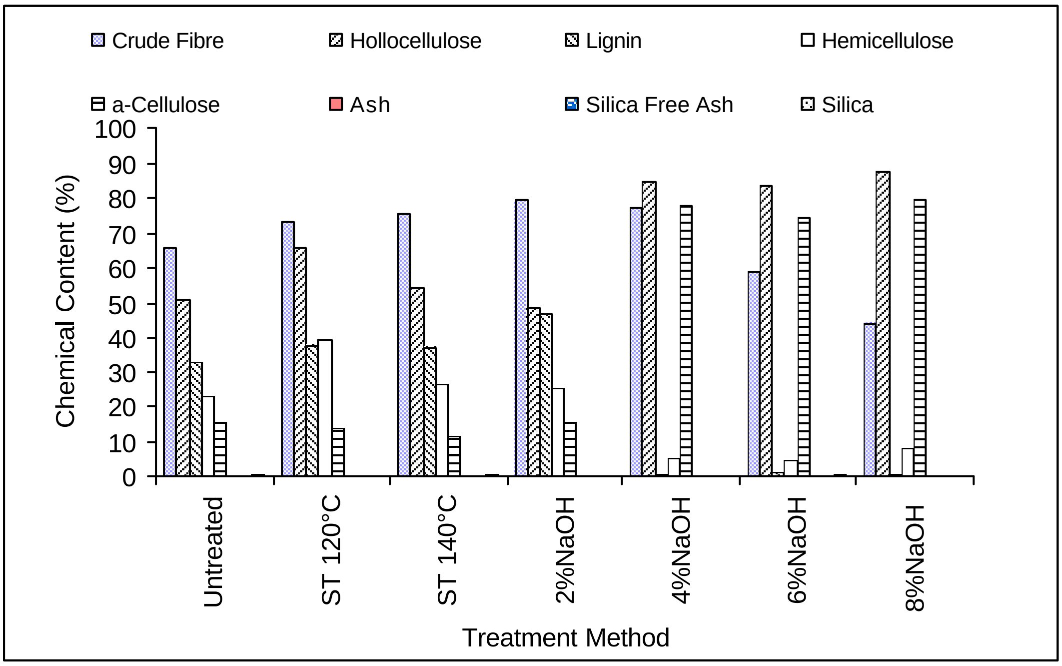

FIGURE 6- EFFECT OF STEAM CURING AND ALKALI TREATMENT ON CHEMICAL CONTENT OF RICE HUSKS

Thermal Stability of Alkali Treated Rice Husks

Figure 7 presents the effect of alkali treatment on thermal stability of rice husks. Initial weight loss of 3.5%, which involved removal of water from the samples between 61 and 150°C, was not dependent on the treatment. This weight remained almost stable from that temperature up to 218°C where substantial thermal degradations of untreated and alkali treated rice husks were noted. Degradation of alkali treated rice husks in this stage was continuous from 218 to 362°C. However, degradation of untreated rice husks was slightly different from that of alkali treated rice husks. The main difference was the presence of an intermediate weight loss of 20% between 218 and 334°C (Table 1), which did not occur in alkali treated rice husks. It was also observed that the degradation of untreated rice husks occurred at higher temperatures, whereby its corresponding inflection temperature was found to be 32°C higher than that of alkali treated rice husks. The final degradation temperature of untreated rice husks was also 14°C higher than those of alkali treated rice husks. The decrease of the thermal stability of alkali treated rice husks was due to removal of certain components of rice husks. The result of chemical composition analysis in Figure 6 showed that the components, which were removed from rice husks during alkali treatment, were lignin and hemicelluloses. Based on this observation, the decrease of the thermal stability of alkali treated rice husks should thus be associated with removal of lignin and/or hemicelluloses from rice husks. However, according to Rowell [19], lignin has the highest thermal stability among the three components (cellulose, lignin, and hemicelluloses) and thus degrades at higher temperatures. The decrease of the thermal stability of alkali treated rice husks was due to removal of lignin and not hemicelluloses. Absence of intermediate degradation stages between 218 and 334°C in alkali treated rice husks was due to elimination of hemicellulose and volatile components, which degrades in the same range [22]. A different observation has been reported by Rajulu's *et al.* [18] on TGA of *Hildergadia* fabric treated by 5% NaOH**.** They observed in their study that the thermal stability of alkali treated fabrics was higher than that of untreated fabric because of elimination of lignin from the fabric. Their view was that lignin has lower thermal stability than cellulose or hemicelluloses. Its removal from the fabric by alkali treatment is what contributed to the increase of the thermal stability of the alkali treated fabrics. However, Shah *et al.* [21] have demonstrated that the thermal stability of alkali treated lignocellulosic fibres increases probably due to the formation of lignin-cellulose structure. Despite the conflicting opinions presented by various authors [18,19,20], the results obtained in this study show that the decrease of thermal stability of the alkali treated rice husks was indeed due to removal of lignin. The lignin-cellulose structure suggested by Shah *et al.* [21] was probably not formed in this case. Possibly, the decrease of thermal stability of *Hildergadia* fabric reported [18] was due to removal of hemicelluloses [17]. It can also be shown in Figure 7 that the residue (char) of untreated rice husks was 8% higher than that of alkali treated rice husks after heating the samples to 700°C. The main component of ash is silica. Therefore, the decrease of residue char content, which contains ash, in alkali treated rice husks may be related to partial removal silica during alkali treatment.

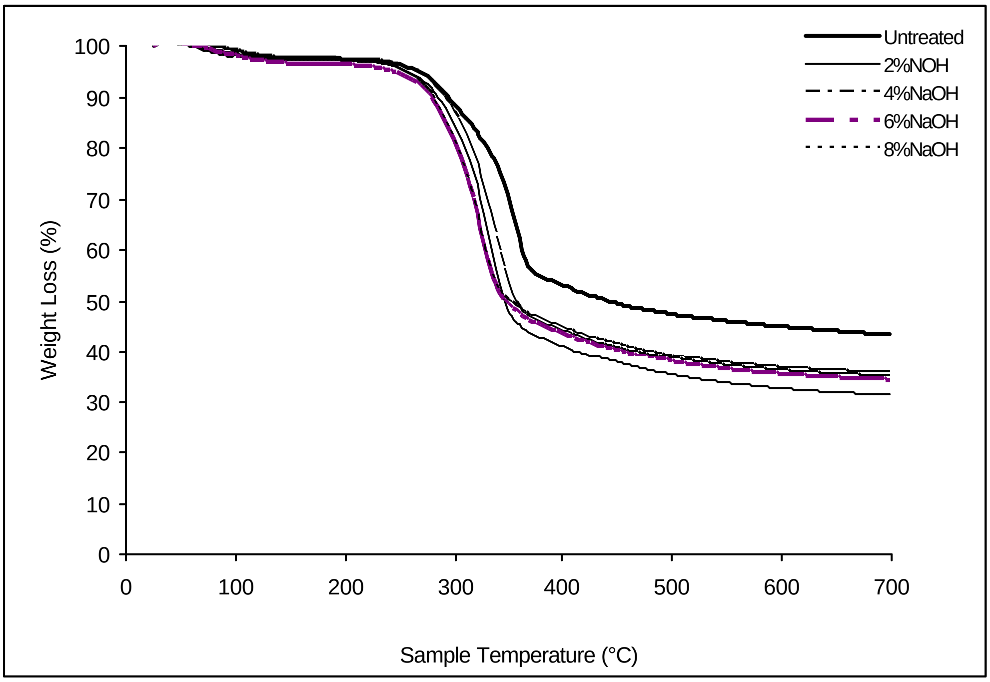

FIGURE 7- TGA THERMOGRAMS OF UNTREATED AND NaOH TREATED RICE HUSKS

Thermal Stability of Steam Treated Rice Husks

The results of TGA thermograms of steam cured rice husks are presented in Figure 8 and in Table 1. As can be observed in Figure 8, that steam curing of rice husks from 100 to 140°C did not cause significant effect on the thermal stability of the rice husks. The weight loss characteristics of steam cured and untreated rice husks with increase of temperature were similar. Although steam treatment and steam explosion are reported to cause remarkable chemical and physical changes of the cell wall components [10, 12], the temperatures applied in this study seem to be low enough to cause substantial change on the properties of rice husks. This is indicated by similar degradation patterns of untreated and steam treated rice husks and equal amount of residue left after heating all the samples to the temperature of 700°C (Figure 8). The only change was slight decrease of the initial degradation and intermediate temperatures for rice husks cured at 140°C (Table 1). Thus, steam curing of rice husks up to 140°C does not cause significant change of the thermal stability of the rice husks.

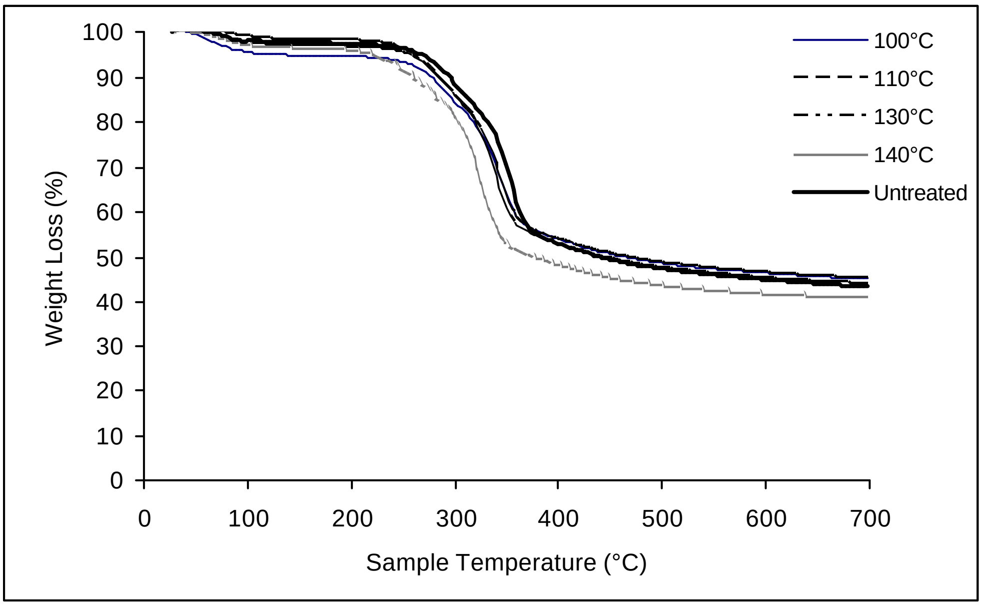

FIGURE 8 - TGA THERMOGRAMS OF UNTREATED AND STEAM CURED RICE HUSKS

TABLE 1 - DEGRADATION TEMPERATURES OF UNTREATED AND NaOH TREATED RICE HUSKS.

		Parameter (°C)				
		Initial instability	Initial degradation temperature	Intermediate Degradation	Inflection temperature	Final degradation temperature
Untreated		61-120	218	218-334	356	376
Alkali treated rice husks	1% NaOH	61-120	218	-	324	362
	2% NaOH	61-120	218	-	324	362
	4% NaOH	61-120	218	-	324	362
	6% NaOH	61-120	218	-	324	362
	8% NaOH	61-120	218	-	324	362
Steam cured rice husks	100°C	61-120	218	218-334	345	356
	110°C	61-120	218	218-334	342	356
	130°C	61-120	218	218-334	339	356
	140°C	61-120	197	197-313	323	358

Effect of Rice Husks Degradation on the Mechanical Properties of Particleboard

Figure 9 shows the relation between chemical degradation of rice husks and internal bond strength of particleboards manufactured from steam cured and alkali treated rice husks. The decrease of crude fibres content in rice husks with increase of the concentration of NaOH was proportional to the decrease of internal bond strength of the particleboard. A similar trend is shown by lignin although the relation is not precisely the same as that of crude fibres. Thus, chemical degradation of alkali treated rice husks has a negative effect on the internal bond strength of the particleboards.

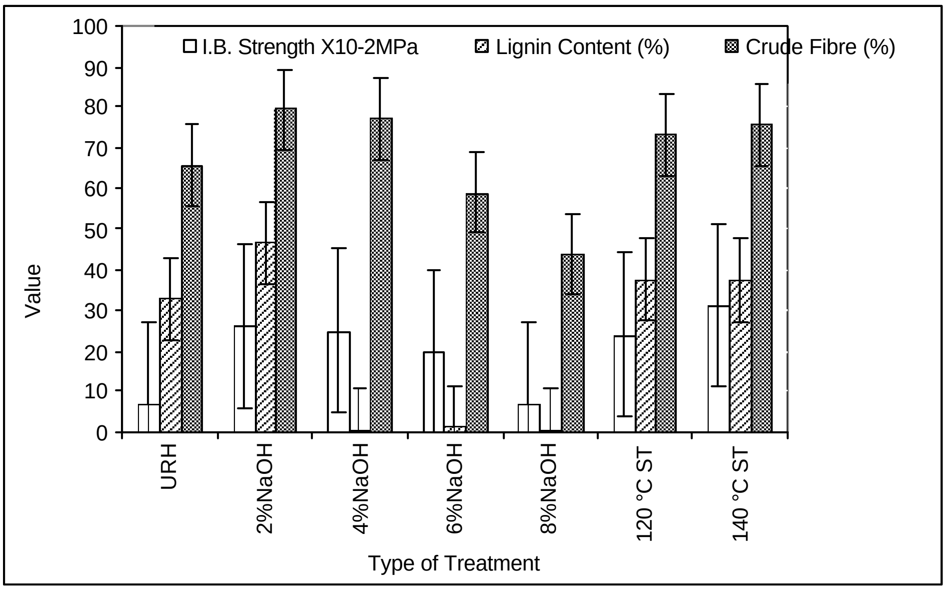

FIGURE 9- INTERNAL BOND STRENGTH AND LIGNIN CONTENT RELATION

CONCLUSION

Alkali treatment of rice husks affects the chemical composition, physical properties and thermal properties of rice husks. The effect becomes more profound as the concentration of NaOH increases beyond 4%. Treatment of rice husks with alkali at the concentration of NaOH exceeding 4% reveals significant degradation of lignin and hemicellulose. The outer surface of rice husks undergoes significant physical degradation when treated by alkali with NaOH concentration higher than 6%. Removal of lignin was responsible for the reduction of thermal stability of rice husks. Decrease of the residue by 8% at 700°C in alkali treated rice husks indicates that part of the silica decomposed although chemical composition analysis did not reveal this.

Steam treated up to 140°C does not affect the physical properties, chemical or even thermal properties of rice husks the same way alkali treatment did. Thus, in order to produce significant physical and chemical changes of the properties of rice husks the steam temperatures should be raised to above 140°C.

Chemical degradation of alkali treated rice husks has a negative effect on the internal bond strength of the particleboard.

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