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Case study

Formaldehyde free particleboards from wood chip wastes using glutaraldehyde modified cassava starch as binder

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ABSTRACT

The study determined some physical and mechanical properties of particleboards produced from wood chip wastes and modified cassava starch (MCS). The native cassava starch was modified with 25% glutardialdehyde solution. Density, water absorption (WA), thickness swelling (TS), modulus of elasticity (MOE) and modulus of rupture (MOR) were evaluated based on Japanese Industrial Standard (JIS). Scanning electron microscope (SEM) Fourier-transform infrared spectroscopy (FTIR) were utilised to conduct the microstructural and elemental analyses of the particleboards. Density ranged from 0.21 to 0.54 g/cm^3, WA ranged from 32.7 to 168.9% after 2 and 24 h immersion while TS ranged from 1.8 to 67.9% after 2 and 24 h immersion. Peak values at 3232.0 N/mm^2 and 35.7 N/mm^2 for MOE and MOR were recorded for the MCS bonded boards. SEM showed well-dispersed MCS granules on the surface of the particleboards while FTIR indicated the occurrence of aldehyde and ester forms attributed to MCS binder. The modified cassava starch bonded boards showed good prospects for utilization in non-load applications having displayed a better performance than the unmodified particleboards.

1. Introduction

Huge quantities of post-consumer waste timber are generated worldwide at a significant rate and the level of its recycling is small when compared with other construction and demolition wastes. It has been reported that between 8 and 50% of the entire wood-based material being utilised yearly for various applications become wastes. A typical wood waste consists of solid timber, timber shavings, sawdust, veneer panels and combinations of two or more of them [1]. Wood wastes from the construction and demolition locations majorly originate from ceilings, flooring and decorations and it accounts for more than 10% of the total solid waste from that sector alone with exception of concrete waste. They are mostly dumped in landfills or burnt in a combustion chamber [2]. However, these wood wastes could be reused by sorting, processing and utilised as raw material for the development of particleboards and fibreboards. These could be used as ceiling panels, flooring, wall and office dividers, furniture, cabinets, bulletin boards, and desk tops. Further reutilization of the recycled wood wastes could lead to a higher release of emissions from the products within an enclosed environment than the expected dose. These could be from two sources, the first is due to incompletely reacted formaldehyde-based resins from the recycled wood waste; while the second could probably come from the new formaldehyde binder utilised in forming the new product [2]. Based on

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these factors, the indoor air quality of most modern buildings could deteriorate and pose serious health challenges for its occupants' more than older structures which are less airtight and are free from products from recycled wood waste. This possible health problem further showed the importance of the role of formaldehyde in inducing illness through immune mechanisms and direct toxicity. Therefore, to overcome this problem various researches had been concluded on how to reduce formaldehyde emissions from the wood composite panels. Two approaches had been pursued, the first method involves the reduction in the amount of formaldehyde resin used in the production of the boards through chemical modification [3]. Such studies include [4–10]. The second method involves the use of formaldehyde-free adhesives, these materials are green, environment friendly and could be used to produce particleboards without the aforementioned health issues. Among such are natural latex [11], soy-based adhesives [12,13], gum Arabic [14], wheat gluten [15], corn starch [16] and nano modified starch [17] amongst others. Starch is one of the richest sources of natural polymers being a low-cost material. Grains, tubers and roots (cassava) are some crops in which commercial quantities of starch for industrial uses are found. It comprises of amylose and amylopectin which are known by their chemical structure as shown in Fig. 1.

Africa is the biggest producer of cassava in the world with Sub-Saharan Africa having the largest share with 55% while Asia also contributes 30% to the value [19]. It's a source of food in Africa while it is used in industries in Asia and it has been a major raw material for various products on a large scale without affecting its use as a food source and the ecosystems. This is based on the long-term observation of the fact that cassava thrives in poor fertile soils unlike other sources of starch [20]. As previously highlighted, starch has been successfully used as resins in wood-based industries. But, the main drawbacks of such starch bonded boards are the dimensional stability and poor bonding properties which are more pronounced in cases involving starch as adhesives only mainly due to its hygroscopicity. Nevertheless, certain treatments have been utilised in some studies to modify and improve the use of starch as binders in different applications. Such studies are [21,17]. Some studies exist already on utilising chemically modified starch as resin in particle board production. Chotikhun and Hiziroglu [22] evaluated properties of particleboard panels manufactured from Eastern redcedar using modified starch and low percent of urea formaldehyde as binder. The results indicated that higher values of MOR, MOR and density were observed in samples with the modified binder in comparison with the unmodified ones. Amini et al [23] similarly developed composite boards using rubberwood bonded with modified starch and reported that the modulus of rupture and the internal bond strength of the panels fulfilled the condition of the standard. The main goal of starch modification is to bring in some functional groups to improve its role in various applications. The major chemical modifications are radical graft copolymerization, substitution reaction (esterification, etherification), crosslink reaction and oxidation reaction. Very limited studies are available on reuse of woodchips from furniture and wood processing activities in the development of particleboards. Similarly, few experiments exist on utilization of cassava starch obtained as a waste from cassava processing and their subsequent modification, being used with woodchip wastes in the same system for the production of particleboards. Therefore, the present work aims to bridge this gap through their use in development of wood particleboards.

2. Materials and methods

Wood chip wastes (WC) were sourced from furniture industries around the vicinity of Landmark University and sorted to remove unwanted debris and non-wood substances. The WC was dried in a Mermmet oven at 60°C until consecutive readings on the moisture analyzer were ±3% moisture content (dry basis) and thereafter milled into particles. The WC particles were sieved and separated into two different sizes of 0.85 mm and 1.7 mm using standard sieves. Cassava starch (SA) was procured from a commercial outlet in a powdered form and mixed with glutaraldehyde in liquid form in a ratio of 1:2 (w/w) based on recommendations from [4]. Glutardialdehyde solution is an oily liquid and colourless which is used in health facilities was supplied by Tianjin Kermel chemical reagent Co., Ltd. It should be noted that the cassava starch is usually a by-product waste material obtained during cassava processing in form of wastewater which is processed further to get the powder form and are available for commercial purposes. SA powder was dissolved in distilled water at 23 °C and stirred manually for about 40 s. 25% glutardialdehyde solution was added to the solution based on recommendations of Amini et al [23] and stirred for 2 min. at a temperature of 23 °C to form the modified cassava starch (MCS). Fig. 2 showed the chemical structure of starch that has been modified by glutaraldehyde.

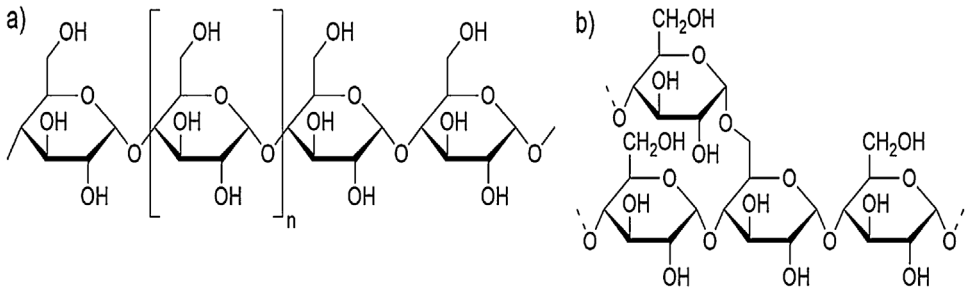

Fig. 1. Molecular structure of (a) amylose and (b) amylopectin [18].

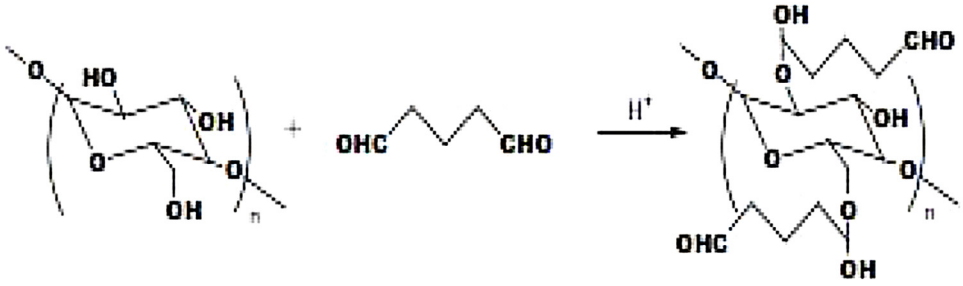

Fig. 2. Schematic diagram of modified starch by glutaraldehyde (MCS).

Two different dosages of the MCS adhesive of 1.5 and 2.5 based on mass ratio were used as shown in Eq. (1).

$$\text{Dosage, } D = \frac{M_{resin}}{M_{woodwaste}} > 1 \tag{1}$$

Where M_{resin} is the mass of resin, $M_{woodwaste}$ is the mass of the sawdust.

Eight different panels of nominal dimensions of 610 mm × 400 mm × 35 mm (thickness) were produced (Fig. 3). The mix design is shown in Table 1. A measured quantity of WC was manually mixed with both SA and MCS based on the composition and cold pressed into a mat under 2.5 MPa for 24 h. Afterwards, they were hot pressed at a pressure of 3 MPa and using temperature of 150 °C for 3 h and thereafter conditioned in a controlled environment with a temperature of 23 °C and a relative humidity of 60% for ten days. Five samples of 150 mm x 50 mm x 35 mm were cut from each board and mechanical tests were performed on them. The test samples were conditioned as reported by [24] until variations of 0.2% were achieved on the test pieces after two consecutive weighing actions within 24 h. After the conditioning procedure, the mechanical tests conducted were the modulus of rupture (MOR) and modulus of elasticity (MOE) in accordance with Japanese standard [25]. Both tests were conducted on a Testometric Universal Testing Machine (Model No M500-50AT) using crosshead speed of 10 mm/min.

Water absorption and thickness swelling tests were carried out in accordance with Japanese standard after 2 and 24 h immersion in water. Water absorption (WA) was calculated according to the formula:

$$\text{Water absorption } (\%) = \frac{W_f - W_b}{W_b} \times 100\% \tag{2}$$

Where W_f is the sample weight after a period of immersion in water and W_b is the oven-dry mass of the sample. Thickness swelling (TS) was calculated from the differences in a sample's thickness before and after soaking in water using a digital vernier calliper of precision of 0.01 mm. TS percentage was calculated using the formula:

$$\text{Thickness swelling } (\%) = \frac{T_f - T_i}{T_i} \times 100\% \tag{3}$$

Fig. 3. Wood chip particle boards.

Table 1
Particleboard mix design.

Particleboards	Wood waste particle size (mm)	Dosage of binder	Type of resin
R	0.85	1.5	MCS
I	0.85	1.5	SA
D	0.85	2.5	MCS
W	0.85	2.5	SA
A	1.7	1.5	MCS
N	1.7	1.5	SA
G	1.7	2.5	MCS
B	1.7	2.5	SA

Where T_f is the final swelling and T_i is the initial swelling. Density was calculated on the assumption that water used for immersion has a density of 1 g/cm^3 using the formula:

$$\text{Density}\,(\text{g/cm3}) = \frac{W_b}{V_w} \tag{4}$$

Where W_b is the oven-dry mass of sample and V_w is the volume of the specimen. The mean results of three test pieces from each particleboard were calculated to achieve higher precision. The fractured samples were polished and thereafter examined using a field emission scanning electron microscope (FESEM) (model: JSM-7600 F, Jeol, Japan) at an acceleration potential of 15 kV and a magnification of 10,000 and 12,000. The polished surfaces of the specimens were sputter-coated with a thin layer of platinum using a JFC-1600 auto fine coater. FTIR spectra were used to determine the functional groups of the samples using a Shimadzu FTIR- 8400S spectrophotometer. The spectra for the specimens were recorded by grinding the specimens to powder, mixing the powder with a small amount of potassium bromide powder and compacting the mixture into a disk. Standard deviation and One Way ANOVA were used to analyze the results.

3. Results and discussions

3.1. Dimensional stability and density

Starch being a hygroscopic material still maintained its peculiar traits even after modification by glutaraldehyde thereby affecting the dimensional stability of the samples. It is observed in Table 2 that water absorption of samples bonded with MCS was lower in comparison with samples bonded with SA. WA ranged from 32.7 to 140.7% after 2 h in water while it ranged from 33.8 to 168.9% after 24 h immersion in water. Groups with same particle sizes showed 137% WA reduction at sample R when compared with sample I, sample D had 18.5% reduction in comparison with sample W, 123% reduction in sample A in relation with sample N and sample G had 150% reduction in water uptake when compared with sample B after 2 h immersion in water with significant differences recorded. Similar traits were observed for WA after 24 h in water which indicates that the MCS was able to improve the chemical structure of the panel by creating films which acted as a barrier against water movement through capillary action in the internal network of the boards. Another reason for this improvement is that modifying the native starch with glutardialdehyde led to the development of distarch glycerols which is made up of linkages between the cross-links and the hydroxyl groups of the starch and this subsequently produced the more water resistant traits observed on the usage of the MCS binder [26].

However, the results did not satisfy the minimum requirement reported in the JIS standard but this could be solved by either coating the wood fibres with water repellent chemical prior to board manufacture or through coating the surface of the finished board with the same chemical [27]. A similar observation was made by Amini et al [4] when it was reported that corn starch-modified with glutardialdehyde performed badly in comparison with the JIS standard. Samples bonded with

Table 2
Physical properties result.

Particle boards	Density (g/cm^3)	2 hrs W.A. (%)	24 hrs W.A (%)	2 hrs T.S. (%)	24 hrs T.S. (%)
R	0.44(0.03) ^{a}	32.7(5.3)^{a}	81.3(5.2)^{a}	9.0(0.6) ^{a}	15.6(1.8) ^{a}
I	0.21(0.01) ^{b}	76.2(2.7) ^{b}	33.8(2.7) ^{b}	1.8(0.04) ^{a}	2.6(0.03) ^{b}
D	0.54(0.02) ^{a}	92.8(5.9) ^{c}	100.5(9.3) ^{b}	59.4(1.0) ^{b}	67.9(2.1) ^{c}
W	0.47(0.01) ^{c}	109.9(5.9) ^{b}	115.3(6.8) ^{c}	24.2(1.4) ^{a}	35.5(1.6) ^{d}
A	0.44(0.01) ^{d}	38.3(3.4) ^{d}	52.9(2.1) ^{d}	3.0(0.05) ^{a}	4.8(0.09) ^{e}
N	0.28(0.02)^{e}	85.7(3.2) ^{b}	104.1(4.5) ^{e}	12.3(1.7) ^{a}	15.4(1.3) ^{b}
G	0.52(0.03) ^{f}	56.8(3.3) ^{e}	82.1(3.2) ^{f}	13.4(0.9) ^{a}	23.1(2.8)^{f}
B	0.30(0.01) ^{g}	140.7(5.3) ^{b}	168.9(3.0) ^{g}	8.4(0.03) ^{a}	12.6(0.4) ^{b}

Values in parenthesis show standard deviation while different letter shows significant difference between groups within the same columns at $\alpha = 0.05$.

MCS had similar pattern of WA for TS which ranged from 3.0 to 67.9% while SA bonded boards ranged from 1.8 to 35.5% as a result of soaking in water after 2 and 24 h. The reason for this improved performance was because the glutardialdehyde modified starch was able to crosslink with the functional group of the wood chip waste [28]. This was caused by the gross penetration of MCS which is the movement of the adhesive from the outer surface into the capillary network of wood fibres and in the process encapsulating the cracks and debris formed during the development of the boards. This movement is caused by capillary action and hydrodynamic flow. The hydrodynamic flow commences when the hydraulic press is used to fuse the wood particle surfaces to be bonded together. MCS then flows into the interconnected structure of lumens and pits with movement majorly along the path of least resistance [29]. It is worth mentioning that despite the poor dimensional stability shown by samples bonded with both SA and MCS in comparison with the JIS standard, the boards did not disintegrate but was intact after immersion in water for 24 h, thereby preserving the structural integrity of the boards and as such could be used for both indoor and outdoor applications [15]. Density ranged from 0.28 to 0.54 g/cm^3 with the smaller particle size (0.85 mm) having the higher value in comparison with another particle size (1.7 mm). Most of the best results were from boards bonded with MCS at 1.5 and 2.5 dosages due to increased surface contact between wood fibre which led to the increase in Van der Waals forces. These factors contributed to making the boards become more compact and closer to each other leading to improved mechanical strength. It has also been postulated that density is closely related to the mechanical strength of boards most especially the bending strength [30]. It could also be seen that high-density boards bonded with MCS had low TS and WA in comparison with low-density boards made from SA which had higher TS and WA. The particleboards could be used for interior purposes in residential and agricultural buildings as well as for decorative purposes.

3.2. Mechanical strength

The highest mean results for M.O.E and M.O.R are 3232.0 N/mm^2 and 35.7 N/mm^2 respectively as shown in Table 3 which are for sample G with MCS adhesives. It could be observed that particleboards bonded with MCS displayed the best mechanical strength because the glutardialdehyde was able through its aldehydic groups react with the starch granules and the hydroxyl group of the wood particles. This was possible because the hydroxyl groups of the wood chip wastes are capable of undergoing self-polymerization and cross-linking reaction with the aldehydic groups [31,32]. This cross-linking reaction led to the development of starch that is resistant to mechanical shear and it also enhanced the bonding between the wood fibres and the starch granules. In another study on the use of modified starch as binder in particleboard development, Imam et al [33] reported that despite wood fracture during the mechanical test of particleboards, the adhesive joint in the experiment was intact which meant that superior bond strength was established which was more than the conventional binders such as urea-formaldehyde and phenol-based adhesives used in the wood industry. Samples bonded with MCS such as R, D, A and G had the following MOE and MOR results; 2062.4 and 23.5 N/mm^2, 590.7 and 10.1 N/mm^2, 1885.5 and 19.5 N/mm^2 and lastly 3232.0 and 35.7 N/mm^2. It could be seen that for small fibre size 0.85 mm boards, about 249% (MOE) and 130% (MOR) reduction in mechanical strength was noted on the increase of binder dosage from 1.5 to 2.5 but when the coarse fibre size of 1.7 mm was utilised, an increment in mechanical strength of 71% (MOE) and 83% (MOR) was discovered. This could be interpreted to mean that the MCS could only develop to its maximum potential when coarse sized wood fibre is used to produce particleboards and it is closely related to the high density of the boards as well. An explanation for this situation was that an increased interaction occurred between the particle size and the MCS when the particleboards were compressed. This led to the formation of tighter and more compact boards and it also assisted in obtaining efficient curing of the binder [34]. Samples produced from SA binder are I, W, N and B with MOE and MOR values of 1183.0 and 15.7 N/mm^2, 206.3 and 5.6 N/mm^2, 145.7 and 4.3 N/mm^2 and lastly 353.9 and 14.6 N/mm^2. The mechanical results from SA bonded boards and the MCS boards showed significant differences with MCS boards having better strength performance than the former. Samples R and G met the JIS (2003) minimum requirement of 2000 N/mm^2 for MOE and 8 N//mm^2 for the MOR. All the other particleboards did not perform satisfactorily in relation to the minimum JIS standard. The effective bonding of the MCS to the wood fibre is because of the presence of polar substances on the wood chip surface which increased the

Table 3
Bending test results.

Particle boards	M.O.E (N/mm^2)	M.O.R (N/mm^2)
R	2062.4(321.3)	23.5(2.7) [a]
I	1183.0(217.2) [b]	15.7(1.8) [b]
D	590.7(35.8) [c]	10.1 [c]
W	206.3(12.4) [b]	5.6(0.05) [d]
A	1885.5(198.5) [d]	19.5(2.1) [e]
N	145.7(6.7) [b]	4.3(0.06) [b]
G	3232.0(367.4) [e]	35.7(4.1) [d]
B	353.9(23.8)	14.6(3.5) [b]

Values in parenthesis show standard deviation while different letter shows significant difference between groups within the same columns at $\alpha = 0.05$.

Table 4
Turkey HSD analysis of impact of resin dosages on board properties.

Particleboards	Density	W.A	T.S	M.O.R	M.O.E
R & D	0.3903**	0.0010*	0.0054*	0.0174*	0.0043*
I & W	0.0010*	0.0020*	0.0046*	0.0040*	0.0362*
A & G	0.0040*	0.0210*	0.0018*	0.0053*	0.0078*
N & B	0.0322*	0.0010*	0.0264*	0.0412*	0.0162*

** means insignificant while * means significant at $p < 0.01$ and 0.05.

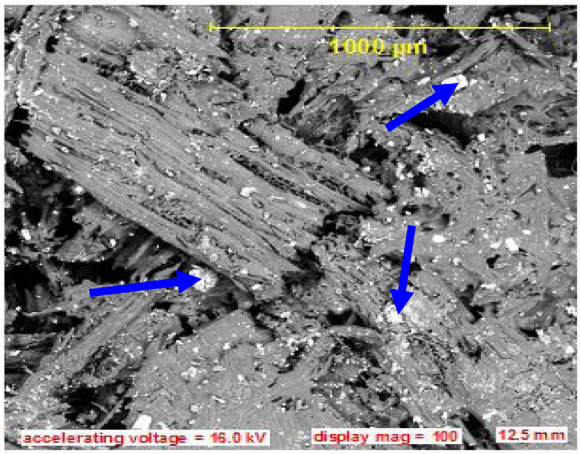

Fig. 4. Micrograph of particleboard with occurrence of glutardialdehyde modified starch granules indicated with blue arrows.

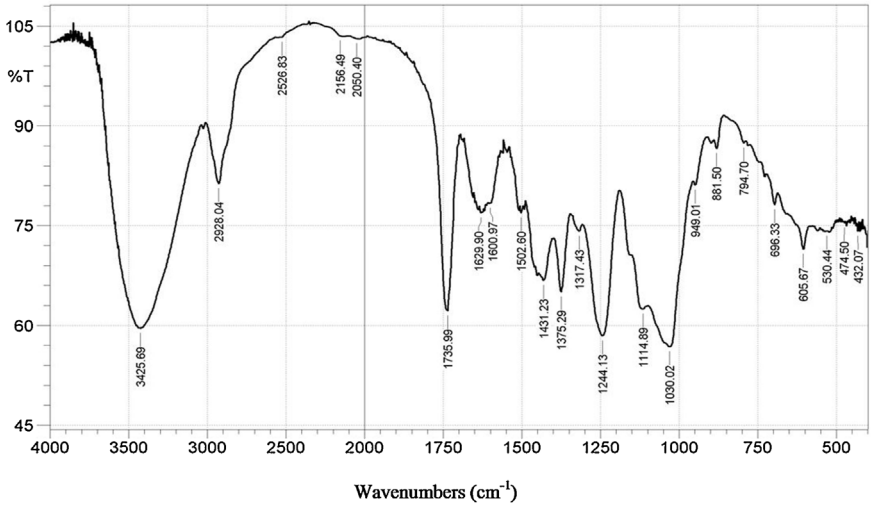

Fig. 5. FTIR characterization of particleboard.

surface energy of the wood and therefore allowed the rapid spread of the adhesive when it came into contact with the solid surface this phenomenon is called wetting. Superior surface energy of the wood encourages better wetting hence improved mechanical strength in samples bonded with MCS [35].

3.3. Influence of resin dosages, particle size and type of starch on particleboards

As the resin dosage was increased in the particleboard, significant differences as shown in Tables 2–4 were observed in majority of particleboards with the same resin type. The Tukey HSD analysis was used to compare boards with same resin type and particle size but different dosages. On increase of resin dosages, particleboard D had 23%

increment in density in comparison with R, sample W had 123% higher density when compared with I, 18% increase in density at G over A and lastly 7% increase in density in comparison with N. The resin dosage increase had significant effect on the density of the particleboards with a slight deviation from boards R and D. The excessive quantity of resin dosages used significantly affected the water absorption, thickness swelling, modulus of rupture and modulus of elasticity as seen in Table 4. This led to significant increase in both the physical and mechanical properties of the particleboards.

Sample R with fine sized wood particles and sample A with coarse sized wood particles had no difference on the density of the tested boards. But relationship between samples I and N which are from fine and coarse wood sizes showed 33% increase in density at sample N. However, when coarse size was used, 4% reduction in density was observed for particleboard G in comparison to board D. Bending strength results showed different wood particle sizes led to both improvement and reduction in the values. Improvements of 447%, 71% in M.O.R. and 253%, 160% in M.O.E. were recorded for particleboards D and G as well as boards W and B. On the other hand, reductions of 9%, 711% in M.O.R. and 21%, 265% in M.O.E. were obtained for particle boards R and A as well as I and N.

The type of starch used also had major impact on the properties of the particleboards. For the density of the particleboards MCS had 100%, 14%, 57% and 73% higher values than SA bonded boards when comparison were made between boards R and I, D and W, A and N and lastly G and B respectively. Significant improvements were similarly observed in the bending strength results obtained among the particleboards. Increments of 74%, 186%, 1200% and 815% were noted for MCS more than SA in the M.O.R results and 53%, 79%, 353% and 144% for MCS more than SA in the M.O.E. results when comparison were made between boards R and I, D and W, A and N and lastly G and B respectively.

3.4. Microstructural analysis

Fig. 4 showed the micrograph of the modified starch bonded particleboard. The presence of numerous whitish granules scattered all over the surface of the sample showed that the glutardialdehyde modified starch granules was consistently mixed with the wood particles thereby leading to the development of well-bonded network over the entire particleboard. The starch granules could be seen to occupy the spaces between the particles and look to be well attached to the woodchip particle surfaces. These factors contributed largely to the improved dimensional and mechanical properties of particleboards bonded with MCS. Although the starch granules were well embedded with the wood fibre, the presence of debonding in some cell walls could be seen which led to the presence of few voids in the particleboards. Insufficient bonding could be caused by the surface roughness from the wood particles and also there is the possibility of improper mixing of the particles which resulted in weak adhesion at the interface [36].

The FTIR spectra in Fig. 5 shows the absorption peaks at 3425.69 and 2928.04cm-1 corresponding to O—H and C—H bond stretching [37]. The FTIR analysis proved the successful modification of cassava starch. The crosslinking of glutardialdehyde with cassava starch resulted into the formation of carboxnyl starch and dialdehyde starch. Chromophores of carbonyl corresponding to the occurrence of glutardialdehyde modified starch as the new link between WC and MCS and also within the starch granules are located at the absorbance peaks of 1735.99 and 1629.90cm^{-1}. These peaks are assigned to aldehyde and esters forms of carbonyl chromophores [3,38,39].

4. Conclusion

This study investigated the possibility of modifying cassava starch powder with glutardialdehyde to reduce its hygroscopic tendency, improve its bonding properties and later used to produce improved and sustainable particleboards. The obtained results showed that wood chip wastes of particle size 1.7 mm bonded with MCS dosages of 1.5 and 2.5 are acceptable to develop the particleboards which met the minimum JIS standard in terms of mechanical strength. However, the dimensional stability tests did not meet the minimum requirement. The modified cassava starch binder had significant differences across all the tests conducted for the boards. It is concluded that the use of modified cassava starch as a binder in industrial production of particleboards should be encouraged to utilize the wastes generated during cassava processing and from wood processing which are in abundant supply in sub-Sahara Africa.

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