

Recycling of Discarded Agro-Plastic Materials into Printed Circuit Bare Boards using Non-Precious Metal Catalyzed Cladding of Electrolessly Deposited Copper

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Abstract:- Consequent end-of-use pollution impact of electrical and electronic items is of increasing concern due to their non-recycleability and toxicity. This has prompted the research for target recyclable and biodegradable alternatives. Here we present, the commutative recycling of rice husk, corn cob and saw dust and plastic (polyvinyl acetate), into biodegradable and environmentally friendly printed circuit unpopulated boards. Employing a pressure of 25000 psi, temperature of 135 °C and using melamine formaldehyde resin as adhesive, particle boards of dimensions 4 cm x 10 cm were produced. The boards were conditioned for 10 days under atmospheric conditions before being used. Surface cleaning, etching and activation with non-precious metal activating colloid prepared with copper acetate and Ammonium Hydroxide. Pottassium Borohydride (KBH₄) was used as the developer prior to electroless copper cladding. The Polyvinylacetate boards were also cut to the same dimensions and treated likewise. Strenght of material analysis of the produced boards, surface morphology and degree of water imbibition were carried out. The electrical conductivity results show that the boards meet acceptable standard values. The rice husk, corn cob and saw dust have tensile of 2.91, 2.60 and 1.92 and bending strenghts of 2.6 KN/mm² to 3.07 KN/mm² respectively. The optimal breaking load of 2.7 KN was obtained with resin concentration of 40%. The surface morphology presents the samples as platelets and forming overlapping surfaces. The surface electrical conductivities are within acceptable ranges and consequently presents these boards as viable alternatives to the synthetic themosets that are commercially available.

Keywords: Corn cob; rice husk; saw dust; particle board; Electroless deposition; Printed circuit board.

I. INTRODUCTION

The search for printed circuit boards (PCBs) dates back to the 19th century, when telegraph, telephone and radio

inventions were being recognized as practical devices for everyday use and they all required wiring connections [1]. The increasingly complex radio circuits demanded alternative wiring technology which needed to be simpler than the existing tedious and error prone wiring technology. Albert Hanson in 1903 filed a patent [2] that solved the challenge of multi wire connection and initiated research activities in this area. His patent clearly described the concept of double-sided through-hole circuitry. This first circuit pattern touched on so many concepts that are seen to be of modern origin. Basically five primary function-types exists (Motherboard, Expansion board, Daughter board, Network Interface Card and Adaptor.), based on target utility.

PCBs serve as structural platforms for mounting electrical/electronic units and are required to be non-conductors/dielectrics with dimensional, thermal and chemical stabilities. Desired properties are; high dielectric strength, low dielectric constant, good flexural strength, low thermal coefficient of expansion, high resistance to humidity and inherent high degree of fire retardancy. Consequent to these, plastic have been the choice raw materials for unpopulated PCB production. More so plastics give extended shelf life due to their resistance to corrosion unlike metals [3], they do not require additional finishing steps such as buffing, before plating. Plating enhances plastics' tensile strength, elasticity, and flexural strength while reducing its total coefficient of thermal expansion. Plastics also experiences enhanced abrasion and weathering resistances. Plating on plastics is traced back to the plating of a stannous chloride sensitized celluloid pen parts with electroless silver solution [4].

The current drive for environmentally friendly manufacturing inputs and products motivates the need to identify and commercialize alternative raw material inputs that are easily recyclable, environmentally benign and relatively cheap. On the other hand, precious-metal activation of surfaces presents a manufacturing cost challenge to the industry. Consequently, this work presents the potential of using abundant end-of-use agricultural materials (corn cob, rice husk and saw dust) as substitutes to plastic substrates. Additionally, the adoption of a non-precious metal catalyst approach (electroless plating) in the manufacture of the boards presents a cost reduction opportunity for the manufacture of the PCBs.

The electroless plating approach was first adopted by Brenner and Riddell [5], they described the plating of metallic substrates with nickel or cobalt alloys in the absence of an external source of electric current. Prior to Brenner and Riddell's publication, Wurtz in 1844 [1] reported that nickel cations were reduced by hypophosphite anions but his result gave only a black powder. Breteau in 1911 [2] presented the first electrolessly deposited bright metallic nickel-phosphorus alloys. Roux obtained a patent in 1916 [5] on an electroless nickel plating bath process notwithstanding a poor experimental output. This was the first patent on electroless deposition. Sequel to this, other studies were carried out focused on the chemistry of the process and not on the plating outcome

Until recently electroless plating has been carried out using predominantly Palladium and tin catalysts. This combination has a high cost implication and prompted the search for cheaper alternatives. Studies have been carried out using colloidal solution of cheaper metals like silver, copper and nickel [6],[7] and [8]; colloidal solution of a hydroxide or oxide of a metal such as nickel or copper [9]; inorganic acid and a water-soluble unsaturated organic compound [10]; noble metal salt of methane sulfonic acid as a noble metal colloid [11]. A further effort at cost reduction was carried out by Iwai et al., [12] by using alkali solutions of NiSO_4 , NiCl_2 , CuSO_4 and CuCl_2 and then immersing the immersed objects in a KBH_4 solution so as to reduce the colloids and provide catalytic activity. They also studied colloids of lead, cobalt, cadmium, zinc, manganese and aluminium in addition to nickel and copper. This marked the successful use of KBH_4 and its family members.

In this study, a non-noble pyrophoric metallic particle is employed as catalytic solution in the form of a hydrous oxide. This is feasible because of their pyrophoric nature that allows the formation of metallic nuclei with any oxidized outer surface in contact with it when in an air and water environment.

II. MATERIALS AND METHODS

2.1 Materials

Substrates: - Saw dust, Corn cob, Rice husk and Polyvinyl Chloride (PVC) powder. The sawdust was obtained from a local saw mill in Zaria, and conditioned under shade for 72h, sieved to obtain fine particle sizes of less than 80μ . Corn cobs were collected from local farmers within the locality, ground, sieved and conditioned to a moisture content of not more than 1%. Rice Husk was collected from a local rice mill in Samaru area of Zaria. It was as with the corn cob sample. The treated agro-wastes were pressed into sheets on addition of prepared resin (adhesive).

Polyvinyl Chloride powder was obtained from Hassan Plastics Company, Kano and with necessary additives, pressed into sheets. The sheets were then cut to dimensions.

Reagents: Melamine formaldehyde resin. (Prepared in the laboratory using the method adapted from [13].

Cleaning solution; Sodium hydroxide and Methanol; obtained from BDH Poole London.

Etchant (PVC board only), Chromium oxide and Tetraoxosulphate (vi) acid (from BDH Poole London). Phosphoric acid (procured from Sigma-Aldrich GmbH Germany).

Catalyst solution (non-precious metal type), Ammonium hydroxide and Copper acetate (procured from Sigma-Aldrich GmbH Germany). Sodium chloride and sucrose (Thermo Fisher Scientific USA).

Catalyst activator: Potassium borohydride (procured from Sigma-Aldrich GmbH Germany)

Plating solution: Copper (ii) sulphate pentahydrate, Sodium potassium tartarate Formalin, Ethylene diamine tetra acetate and Sodium hydroxide; (obtained as analar grade materials from Thermo Fisher Scientific USA).

Equipment and glassware:

Reaction vessel (3 liters), Mechanical stirrer (0-200 rpm), Water-bath (thermostatically operated), kitchen-type blender, Spreader, Carver hydraulic press, Metal moulds (stainless steel flat sheets), Plastic mixing bowls, pH meter, Viscometer (ubbeholde) and Universal testing machine.

2.2 Methodology

Process Flow Chart

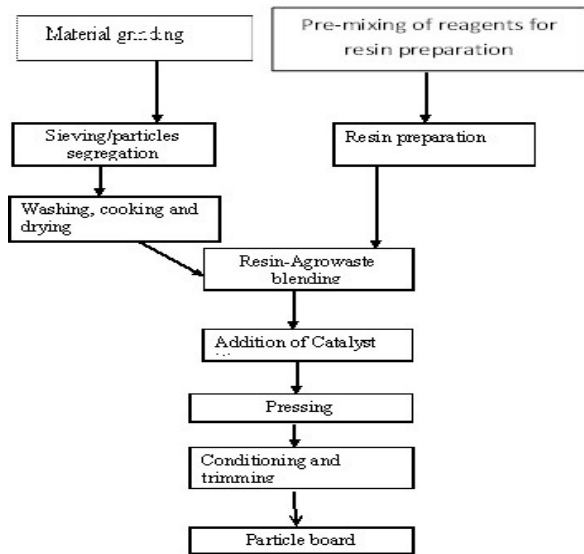


Figure 1: A simplified flowchart of the laboratory production of particle board from agro wastes.

Process method: - Electroless Plating

The steps below depict the stages of treatment: -

- (1) CLEANING ?
- (2) ETCHING (PVC substrates only) ?
- (3) CATALYST APPLIATION (non-precious metal) ?
- (4) CATALYST ACTIVATION (developer) ?
- (5) ELECTROLESS COPPER CLADING ?
- (6) THERMO-TAMPERING ?
- (7) ELECTROPLATING ?
- (8) RINSING and DRYING

Figure 2: Process steps involved in the Electroless and finishing process of copper plating of board samples.

In between steps 1 to 6, rinsing is carried out with deionised water, removing excess reagent preventing unwanted reactions/interactions between steps.

2.3 Analysis and characterization:

2.3.1 Adhesion Test

A simple test to ascertain the degree of adhesion of the deposited copper metal was done by the use of masking tape. The tape was placed on the surface of the copper-clad substrate and then peeled to evaluate the degree of detachment of the copper coating on the substrate.

2.3.2 Strength of Material Test

The tensile and bending strengths of the samples were done using Micro Vision universal material testing machine. The results of the test influenced the selection of the best blend ratios for the substrates used in the production of the boards used.

2.3.2.1 Bending Strength (Modulus of Rupture)

Three specimens of 180 mm x 45 mm x 2 mm from each sample were tested using three-point bending method (plate 7). The distance between the points is 100 mm and load applied at the centre by the universal testing machine. The speed of tests on this machine was 50mm/min. The results were obtained using the following formula given by the standard:

$$S = \frac{3pd}{2bt^2}$$

Where, S = Bending Strength in N/mm²

P = Load at rupture in Newton (N)

d= Distance between support in Millimetre (mm)

b = Width of the test specimen in Millimetres (mm)

t = Thickness of the test specimen in Millimetres (mm)

The critical stress value to which the material is subjected is the tensile stress along the bottom face - referred to as the board Stress. The formula relating the stress to applied load is:

$$\text{Stress } (\delta) = \frac{3WL}{2bt^2}$$

Where W = load (force) applied at mid-point

L = distance between supports

b = bar with

t = bar thickness

At the point of failure, δ becomes the ultimate flexural stress (also called flexural strength

or cross breaking strength)

Strain is given by: $\delta = \frac{v}{t/L^2}$

L=distance between supports

T=thickness of bar

Y=deflection at the mid-point.

2.3.2.2 Tensile Strength (Parallel to Surface)

Three specimens from each sample were tested for tensile strength test. The effective dimensions of each specimen

were 34mm x 2mm and the lengths were 184mm. The test was performed on a universal Testing machine. The tensile strength was calculated using the formula:

$$T = P/A$$

Where, T = Tensile strength in KNmm-2

P =Maximum Load in KNewton (KN)

A = Cross-sectional area (mm²)

2.3.3 Degree of Water Imbibition

The samples of boards immersed in water for 24 hrs after which the relative amount of water absorbed by the samples found by noting the difference in weight between dry samples and wet samples. The samples also immersed in water and then boiled for 30 mins (boil test) and the relative amount of water absorbed as well as the degree of swelling calculated gave the behaviour of the material in a water environment either in cold or hot condition.

2.3.4 Swelling thickness of each test specimen was obtained by using:

$$G = t_2 - t_1/t_1 \times 100$$

Where, t_2 = Thickness of the test specimen before

Immersion (mm)

t_1 = Thickness of the test specimen after

Immersion (mm)

2.4 Resin synthesis

Method as reported by Crews, Goerge and Shen (1992) in US Patent 5120821 was adopted for the synthesis of melamine formaldehyde resin. The method accommodates the use of impure melamine without compromise to target standard resin product. The synthesized resin had a solid content of approximately 60% (calculated as the relative weight of the residual solid material expressed as percentage of the weight of the resin as introduced and conditioned at a temp of $\geq 105^\circ\text{C}$) and was stable for a period of more than six (6) months without premature cross linking and consequent increase in viscosity. Precisely, the reaction was carried out in an 0.5l thermostatically heated reaction vessel fitted with a reflux condenser and a mechanical stirrer. The pH was constantly monitored after the initial pH of the formaldehyde by the addition of 0.5N aqueous sodium hydroxide. The reacting mole ratios are 1:3 of melamine to formaldehyde and heated to 90 °C. The reaction was complete and terminated when the water tolerance reached 250% at a cook time of 173 minutes and pH of 9.5. At this point 59 grams of ethanol was added to stabilize the product then the product was cooled to room temperature and the viscosity was measured.

2.5 Production of Sheets

The finely sieved agro-wastes (corn cob, saw dust and rice husk), were respectively mixed with the resin at the blend ratio based on prior trials and tensile strength analysis carried out on test samples. The blend of adhesive and agro-waste was moulded and cured at a temperature of 135°C and pressure of 25000 pounds using Carver hydraulic press equipped with thermostatically heated platens (plate 2). The sheets were cut into dimensions of 4cm x 10cm and kept for a period of 10 days to condition. Sheets of PVC were also made and cut to the same dimensions ready for electroless plating.

The cleaning solution was prepared by dissolving 200 g of NaOH in a mixture of 750 mls of deionised water and 250 mls of Methanol. The mixture was stirred and kept for subsequent cleaning of substrate (PVC) at 70 °C for 20 mins (the particle boards were not subjected to this treatment).

The etching solution was prepared by mixing Tetraoxosulphate (VI) acid, orthophosphoric acid and chromic acid in deionised water in a 3:1:1:1 ratio. The etching was done at a temperature of between 40 °C to 70 °C for 2-10 mins. The Chromic acid was prepared by dissolving 30g of Potassium dichromate in 100mls of distilled water and making it up to 1 litre with concentrated (98%) sulphuric acid (the best etching time was determined by a series of trial runs and observed surface texture/appearance).

2.4.1 Activating Colloid:

The non-precious metal activating colloid contained 20 mls (0.25 molar) of copper acetate in 5 ml (0.05 molar) of Ammonium Hydroxide and was used immediately after preparation. Alternative secondary colloids that proved effective when tried are: Gelatine, Glycerol and a solution of 2 g of sucrose dissolved in 15 ml of 1M NH₄OH and mixed with 4 ml of 1 M Copper (II) Chloride.

2.4.2 Developer: 0.1 g/ltr of Potassium Borohydride (KBH₄) was used as the developer.

2.4.3 *Electroless Copper bath*: Three different bath compositions were tried and the most suitable selected for plating on the particle boards. The three compositions are:

Table 1: Composition of Electroless baths used

Reagent	Quantity(g/l)	Quantity(g/l)	Quantity(g/l)
Copper sulphate	15	2.5	12
Formalin	10	3	8
Sodium Hydroxide	15	6	15
Sodium Potassium Tartarate(Rochelle's salt)	10	-	14
Ethelenediamine tetraacetate(EDTA)	20	2.2	20

III. RESULTS AND DISCUSSION

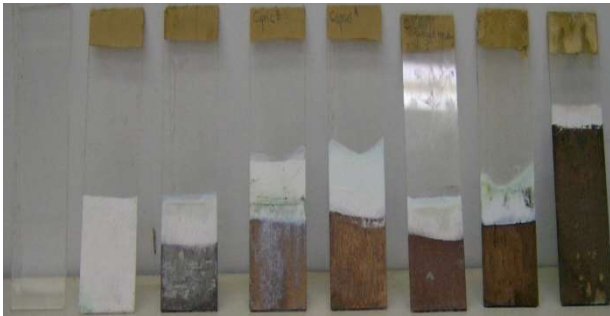


Plate 1: PVC boards arranged in the order of treatment from left to right, unclad to clad.



Plate 2: Corn cob boards arranged in increasing order of treatment from unclad to clad.



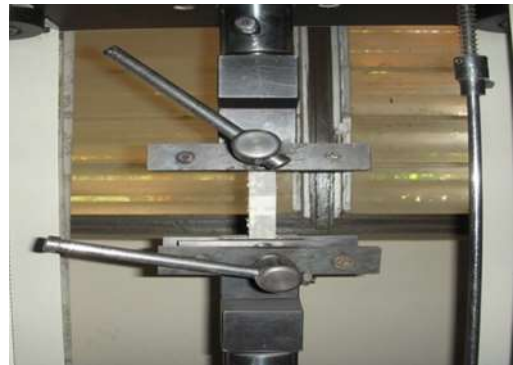
Plate 3: Saw dust boards arranged in increasing order of treatment from unclad to clad



Plate 4: Virgin unpopulated printed circuit board with circuit patterns sketched.

3.1 Strength of Material analysis

3.1.1 Tensile and Bending strengths



Plates 5 and 6: Applied methods for Tensile and bending strength analysis

The test results of tensile strength perpendicular to the surface of the board (Figures 3, 5 and 7) appear to show a consistent relationship between the tensile strength and the board density. The perpendicular tensile strength values for the sample boards increased from 0.084 N/mm^2 for board with 550 kg/m^3 density to about 0.588 N/mm^2 for board samples with 950 kg/m^3 density. The typical mean value specified for wood particle board in the same density range with the experimental boards is 0.34 N/mm^2 (BS 2604, Part 2, 1970) [16]. The perpendicular tensile strength values obtained for boards with densities from 750 to 950 kg/m^3 are above the mean value specified. While the values for boards with density ranges of $550 - 710 \text{ kg/m}^3$ are either below or close to the minimum value established for the wood particle board. Thus, a satisfactory board would have a minimum density of 760 kg/m^3 and tensile strength of 0.34 N/mm^2 and 84.80 N/mm^2 .

Saw Dust

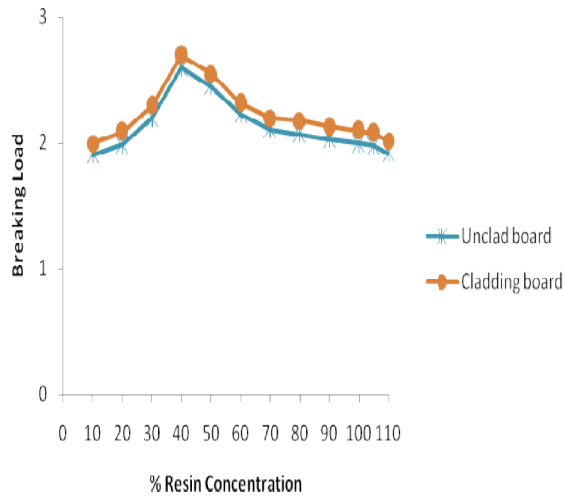


Figure 3: Graph of breaking load for both Clad and unclad board samples against % resin.

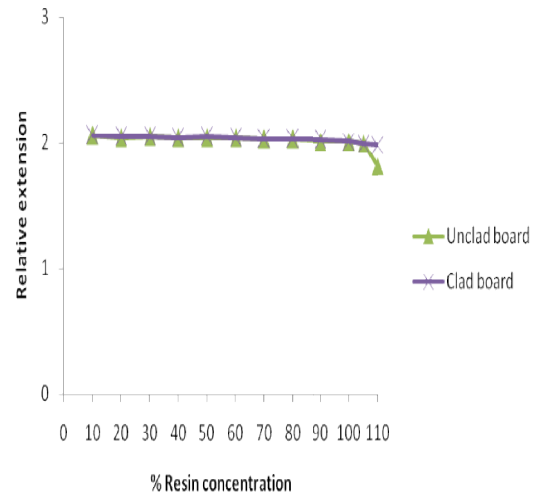


Figure 6: Graph of extensibility of Resin conc. For clad and unclad board.

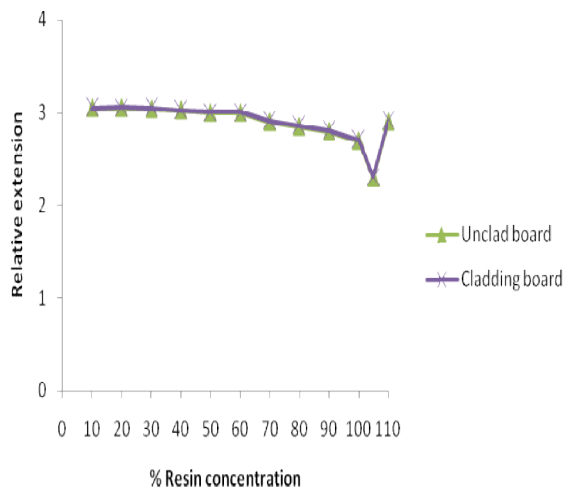


Figure 4: Graph of extensibility of Resin conc. for Clad and unclad board.

Corn Cob

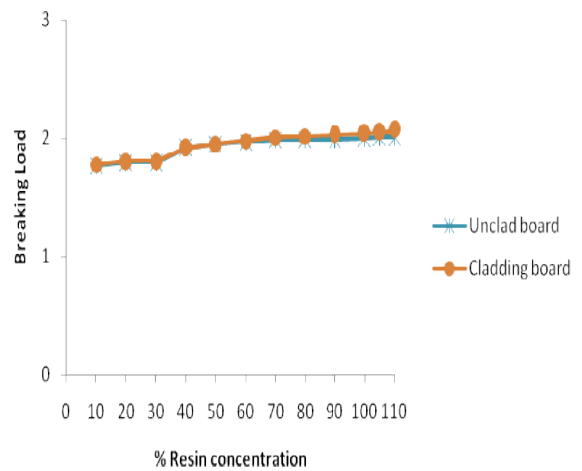


Figure 7: Graph of breaking load for both samples vs Clad and unclad board vs % resin.

RICE HUSK

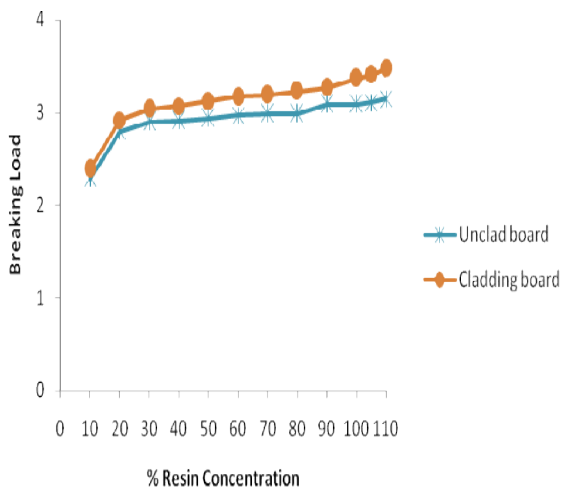


Figure 5: Graph of breaking load for both samples vs Clad and unclad board vs % resin

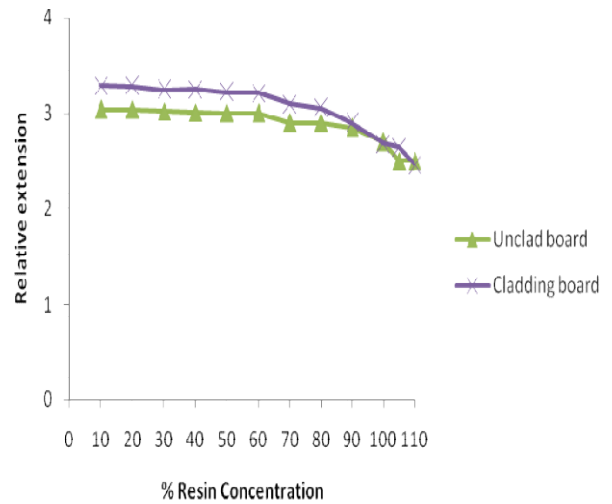


Figure 8: Graph of extensibility of Resin conc. For clad and unclad board

The optimum breaking load value was obtained at resin concentration of 40% for both the clad and unclad boards but with the clad boards having a higher breaking load value of 2.8KN for saw dust (figure 3) but that for rice husk (figure 5) is comparatively higher at corresponding optimal 40% resin concentration. This may be linked to the natural make-up of the waste material from rice husk having silica as its component make up. The silica contributes to the overall strength of the material. The breaking load value for corn cob (both for clad and unclad) followed the same trend but lower than that for sawdust and rice husk samples. The value peaked significantly at 40% resin concentration from which a gradual rise occurred (figure 7). Much difference was not observed between the clad and the unclad samples. The values did not drop even at a resin concentration of 110% as observed with the rice husk samples but differed with that of saw dust.

Figure 9, presents the comparative breaking load values of the copper clad boards from the agricultural materials. Rice husk stands out as having the highest breaking load value as earlier stated.

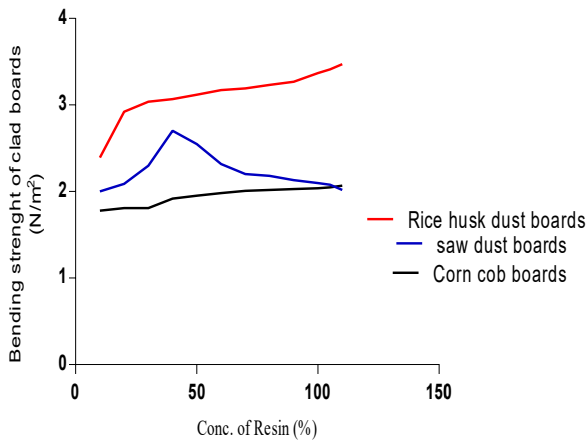


Figure 9: Plot of the clad boards showing the comparative breaking loads against resin concentrations (%).

The result presented as figures 10, 11 and 12 shows the direct relationship between the densities of the boards and bending strength. The relationship in strength values ranged from 2.6 KN/mm² for boards with lower density (kg/m³) to 3.07 KN/mm² for boards with highest density (kg/m³). These values are above the minimum set for wood particle board with average strength ranging from 5.5 to 13.8 N/mm² (BS 2604, Part 2, 1970). It is found also from Figure 10 (by interpolation) that a satisfactory board in respect of this property can be produced from a board density of 1.3 kg/m³. This board would have a minimum bending strength of 2.6 KN/mm².

3.1.2 Moisture absorption:

Moisture absorption is quite a significant property, as the laminate passes through a series of wet process steps, at higher temperatures. The nature of the epoxy resin provides

its tendency to readily bond with available water molecules. Retention of water on the surface reduces the degree of surface insulation, while penetration of water into the bulk of the laminate reduces bulk resistance. It is measured as percentage water retention after immersion of the laminate in hot water for a known length of time. Plots of the degree of impact of resin concentration in relation to board density and amount of water absorbed by board samples made from the different agro materials is displayed as figures 10 to 12 for saw dust, rice husk and corn cob respectively.

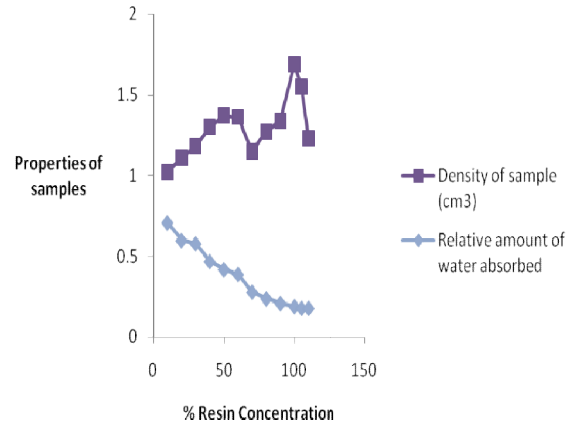


Figure 10: Resin conc. in relation to density and relative amount of water absorbed for saw dust.

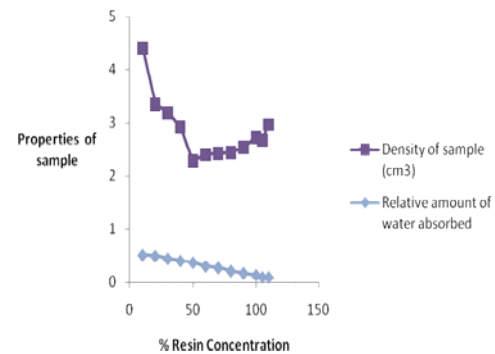


Figure 11: Resin conc. in relation to density and relative amount of water absorbed for rice husk.

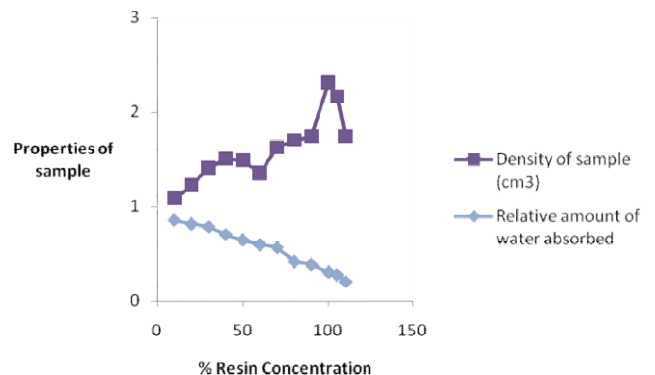


Figure 12: Resin conc. in relation to density and relative amount of water absorbed for corn Cob.

Table II: A summary of correlation between density, tensile strength and relative amount of water absorbed by boards at the optimal 40% resin concentration.

Material	Density @ 40% Resin Conc.	Tensile strength for unclad board@ 40% Resin Conc.	Tensile strength for clad board@ 40% Resin Conc.	Relative amount of water absorbed (g) @ 40% Resin Conc.
Rice Husk	2.93	2.91	3.07	0.41
Saw Dust	1.30	2.60	2.70	0.47
Corn cob	1.51	1.92	1.92	0.71

3.1.3 Surface Morphology

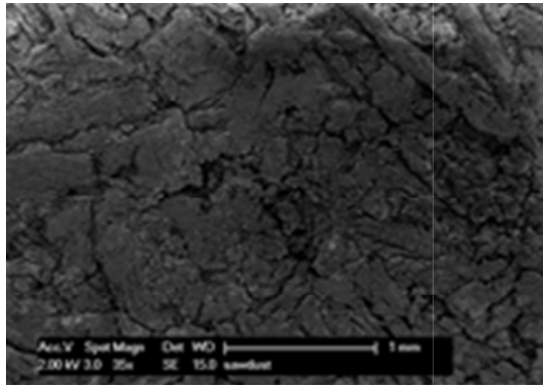


Figure 13 :FESEM of Saw dust board

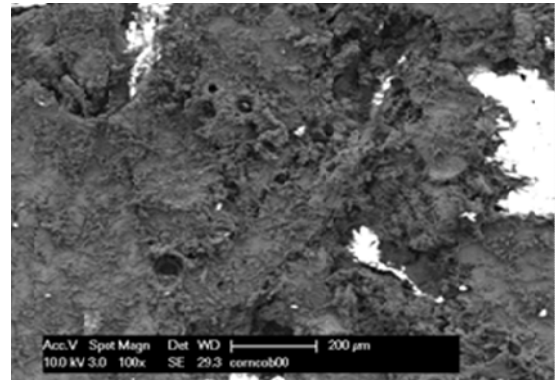


Figure 16: FESEM of PVC board.

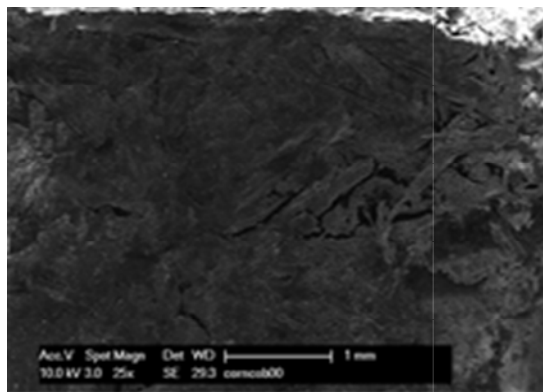


Figure 14: FESEM of Rice husk board.

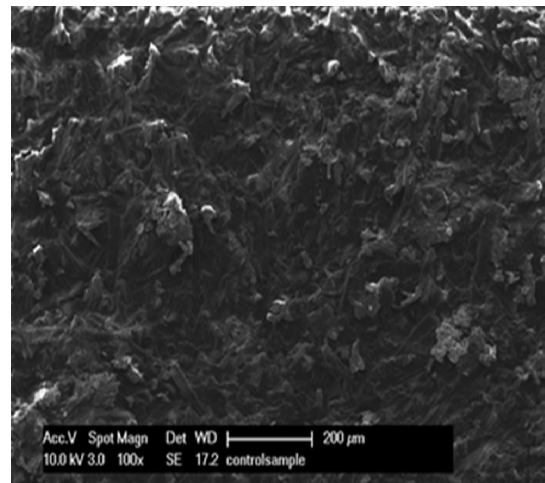


Figure 17: FESEM of control (commercial sample) epoxy board.

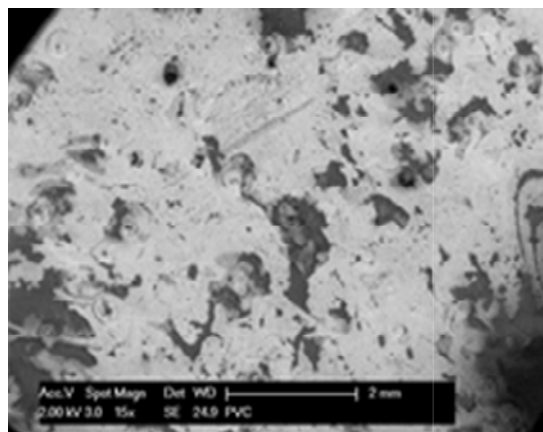


Figure 15: FESEM of Corn cob board.

Figures 13, 14, 15, 16 and 17 are selected spot magnifications of 100x for board samples showing the surfaces to be flat, rough but continuous. Adhesion on the substrate is influenced by the availability of good contact sites (rough contact points). Large increases in surface area promote simple adhesion by providing increased contact sites and corresponding increase in absorbefacient characteristics of the sample. Surface roughness (contact sites) may be enhanced in a variety of processes such as electroplating, electrochemical etching and mechanical etching. Scanning electron microscopy (SEM) allows close inspection of surface morphologies which correlates microstructure and absorbefacient characteristics.

The process of etching imparts on the growth of peak-to-valley distances [14] but does not produce the desired level of roughness and absorbefacient characteristics to samples[15]. This is observed from the morphology of the etched PVC board compared to those of the agri-end-of-use products. The later presents better microstructure characteristics with respect to contact points and their prevalence within the surface areas. Consequently the use of agric-end-of-use materials provides better input material resource in this respect than the synthetic plastic. The use of the waste agro materials with inherent inconsistent surface topology eliminates the manufacturing cost that comes with etching process as well as reduce the production process steps thereby saving production cycle time. The agri-end-of-use samples produced microstructures of small scale roughness and deep V-shaped grooves; which is necessary for capillary flow of solder[16].

IV. CONCLUSIONS

The experiment has shown that agricultural wastes maybe employed as substitute substartes for the production of circuit boards in stead of the synthetic fiber materials. The use of electroless etching conviniently produced the required surface for electrical connection for the application of these boards in the electrical/electronic industry. The overall process presents a feasible environmental friendly alternative route to existing synthetic one. The analysis results showed that the prepared samples meet the required products standards. This work in an overall sense, presents products and methods for achieving a 100% recyclable product manufacturing for the electrical and electroni industry and at the same time provides a means of commutatively recycling and utilization of existing agricultural waste.

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