

Characterization of Breadfruit Seed Hull Ash for Potential Utilization in Metal Matrix Composites for Automotive Application

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Abstract

Breadfruit seed hull (seed coat or seed shell) is an agricultural waste. The waste is produced in abundance globally and poses risk to health as well as environment. Thus their effective, conducive and eco-friendly utilization has always been a challenge for scientific applications. This paper mainly deals with characterization of breadfruit seed hull ash obtained from breadfruit seed hull using spectroscopic and microscopic analysis. The characterization of the breadfruit seed husk ash was investigated through scanning electron microscopy (SEM), X-ray diffractometer (XRD), thermogravimetric analysis (TGA/DTA), Fourier transform Infra red spectrometry (FTIR) and X-ray Fluorescent spectrometry (XRF). Density and refractoriness of the breadfruit seed hull ash were also found. The results were compared and it was observed that the ash possesses nearly same chemical phases and other functional groups as reinforcement like fly ash, rice husk ash and bagasse ash that have been used in Metal Matrix Composites (MMCs) specifically for automobile applications. Hence, breadfruit seed hull ash could be used as a low cost reinforcement in Metal Matrix Composites (MMCs).

Key Words: Breadfruit seed hull, Density; Microstructure, Thermal and Refractoriness

1. Introduction

Researches all over the world today are focusing on ways of utilizing, either industrial or agricultural wastes as a source of raw materials for industrial use. These wastes utilization would not only be economical, but may also result to foreign exchange earnings and environmental pollution control [1-2]. The agro-based industries generate a significant amount of solid wastes with appreciable potential problems. These wastes include peels from plantain, banana and oranges, bran and husk from rice, straw from cereals and hulls from African breadfruit (*Treculia africana*) and soybean [3]. Such wastes if not appropriately handled, could pose disposal problems with consequent effects on the environment and harmonious relationship between the biotic and abiotic components of the ecosystem [6]. The wastes could be disposed of by incineration or by other costly break-down systems or even by illegal dumping, all to the detriment of the environment [3-4].

In the wake of technological advancement, waste accumulation has assumed serious dimensions not

only in the Western World but also in the Third World Countries. Over the years there have been considerable efforts to develop techniques for the recovery and utilization of the biopolymers in the wastes. This became necessary considering the fact that those wastes contain appreciable quantities of dry matter, crude protein, fibre, ether extract, minerals and high molecular weight cellulose and hemicelluloses and could be obtained at minimal cost [1].

Metal matrix composites (MMCs) possess significantly improved properties including high specific strength specific modulus, damping capacity and good wear resistance compared to unreinforced alloys [2-4]. Similarly, there has been an increasing interest in composites containing low density and low cost reinforcements. Among various discontinuous dispersoids used are fly ash, red mud, Rice husk ash [2-5] are some of the most inexpensive and low density reinforcement available in large quantities as solid waste by-product. The African breadfruit tree (*Treculia africana*) is native to many tropical countries like west India, Ghana, Sierra, Nigeria and Jamaica

[6]. One breadfruit pod may contain up to 100-200 seeds. The breadfruit is about the size of a football or more and is greenish in color and the seed hull is brown or black, the edible seed is whitish yellow [7]. The seeds can be dehulled to remove the seed hull (shell) which is discarded as waste. The dehulled seeds are eaten as food, as flour for preparation of biscuits, cookies, loaf and as a potential source of oil [11]. Different avenues of breadfruit seed hull utilization are more or less known but none of them have so far proved to be economically viable or commercially feasible. Hence, the objective of this present work is to characterize breadfruit seed husk in order to explore its use in metal matrix composites.

2. Material and Methods

2.1 Material and Equipment

The raw materials used for this research was African breadfruit seed hull, obtained as wastes from breadfruit processing units market at Awka in Anambra State, Nigeria(Fig. 1)



Fig. 1: Photograph of Breadfruit seed husk

Equipment used in this research are- electrical resistance furnace, X-ray diffractometer (XRD), Scanning electron microscope with energy dispersive spectrometer (SEM/EDS) Machine, X-ray fluorescent XRF, Differential thermal analysis (DTA/TGA), FTIR-8400S Fourier transform infrared spectrophotometer

2.2 Methods

The breadfruit seed hull was dried for 48hrs in the sun, ground to form breadfruit seed hull powder, the powder was packed in a graphite crucible and fired in electric resistance furnace at temperature of 1300°C to form breadfruit hull ash. The ash was ball mill at a speed of 200rpm for 6 hours to obtained fine powder (see Fig. 2).



Fig. 2: Photograph of Breadfruit seed hull ash

Density measurements were carried out on the breadfruit seed hull ash sample using Archimedes's principle. The buoyant force on a submerged object is equal to the weight of the fluid displaced. This principle is useful for determining the volume and therefore the density of an irregularly shaped object by measuring its mass in air and its effective mass when submerged in water (density = 1 gram/cc). This effective mass under water was its actual mass minus the mass of the fluid displaced. The difference between the real and effective mass therefore gives the mass of water displaced and allows the calculation of the volume of the irregularly shaped object. The mass divided by the volume thus determined gives a measure of the average density of the sample [8]. The Pyrometric Cone Equivalent (PCE) as recommended by ASTM Test C-24 was used in the determination of the refractoriness of the sample [9].

Mini Pal compact energy dispersive X-ray spectrometer (XRF) was used for the elemental analysis of the breadfruit seed hull ash. The system is controlled by a PC running the dedicated Mini Pal analytical software [8].

The XRD analysis of the breadfruit seed hull ash was carried out using Philips X-ray diffractometer. The X-ray diffractograms was taken using Cu K α radiation at scan speed of 3^o/min. The samples were rotated at precisely one-half of the angular speed of the receiving slit, so that a constant angle between the incident and reflected beams is maintained. The receiving slit is mounted in front of the counter on the counter tube arm, and behind it is usually fixed a scatter slit to ensure that the counter receives radiation only from the portion of the specimen illuminated by the primary beam. The intensity diffracted at the various angles was recorded

automatically on a chart and the appropriate (r) and (d) values were then obtained [8-9].

The microstructure and the chemical compositions of the phases present in the breadfruit seed hull ash was studied using a JOEL JSM 5900LV Scanning Electron Microscope equipped with an Oxford INCA™ Energy Dispersive Spectroscopy system. The sample was placed on sample holder and the images were captured under various magnifications. Prior to it, sample was applied with the gold coating to avoid charge effect, so to obtain clear images. The SEM was operated at an accelerating voltage of 5 to 20 kV[2].

FTIR-8400S Fourier transform infrared spectrophotometer (SHIMADZU) was used for the functional groups present in the breadfruit seed hull ash. Spectrometer and detector capable of measuring functional group to the predetermined minimum detectable level were employed. The system include a personal computer with compatible software that provides real-time updates of the spectral profile during sample collection and spectral collection using FTIR system at 1 cm⁻¹ resolution, 22 meter path length, and a broad band MCT detector. The Data analysis was performed using appropriate reference spectra whose concentrations can be verified using CTS spectra [10].

3. Results and Discussion

The density of the breadfruit seed hull ash is 1.98g/cm³ which means that breadfruit seed hull ash is very light material. The value obtained fall within the range of density of fly ash, bagasse ash and silica which is 1.8 and 2.2 g/cm³ respectively [2-4] currently used in metal matrix composites. The breadfruit seed hull ash was observed to have Seger Cone No. 22, with equivalent temperature of 1500°C. This means breadfruit seed hull ash can withstand operating temperature of 1500°C without load [2-4].

The XRD pattern (see Figure 3) obtained reveal that, the major diffraction peaks are 53.67, 42.73, 41.40, 31.06, 46.18, 24.31 and 32.59° and their inter-planar distance are: 1.71, 2.11, 2.18, 2.88, 1.92, 3.66 and 2.74Å, and their relative intensity of X-ray scattering are 100.00, 61.90 and 8.44 and phases at these peaks are: iron, Chromium Aluminum Silicon, Aluminum Chromium iron, Potassium Aluminum Sulfate, Sodium Magnesium silicate, Sodium Aluminum Silicate, Aluminum Silicon Carbide, while each of these phases have a score of 42, 30, 29, 34, 21, 29 and 20 respectively (Table 1).

Complete Mineralogical analysis carried out by X-ray diûraction also revealed that the ash contains each of these elements C, O, Na, Al, Si, Cr, Fe, K and none of these elements Br, Kr, Rb, Sr, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I, Xe, Cs, Ba, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At, Rn, Fr, Ra, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf, which means that with the absence of all these other elements the breadfruit seed husk ash may not contain radioactive materials. This is in par with the earlier of other biomass by [2-5].

The XRF chemical composition of the breadfruit seed hull ash is represented in Table 2. XRF analysis confirmed that SiO₂, Al₂O₃, MgO, Cr₂O₃ and Fe₂O₃ were found to be major constituents of the ash. Silicon dioxide, iron oxide, Cr₂O₃ and alumina are known to be among the hardest substances. Some other oxides viz. K₂O, Na₂O and MnO were also found to be present in traces. The presence of hard elements like SiO₂, Al₂O₃, Cr₂O₃ and Fe₂O₃ suggested that, the breadfruit seed hull ash can be use as particulate material for reinforcement of various metal matrixes. This result of XRF is in agreement with the result of XRD obtained. Therefore, the present work suggests the possibility of using breadfruit seed hull ash as particulate in metal matrix composites since the chemical composition has close similarity with the XRF analysis of rick husk ash, bagasse ash and fly ash currently used in metal matrix composites [1-4].

Surface Morphology of breadfruit seed hull ash is seen in back scattered electron (BSE) as shown in Figures 4-5. Breadfruit seed hull ash particles were observed to be solid in nature, but irregular in size. Some spherical shape particles can also be seen in the Figure 4. The chemical analysis of breadfruit seed hull ash morphology consists mainly of Fe, Cr, Si, C, O, Mg, Al as shown in the EDS scan (see Figures 4-5). The results are consistent with XRD and XRF analysis of other biomass by [1-5].

Mainly twenty three peaks were detected in the FTIR Analysis of the breadfruit seed hull ash as visible in Figure 6. These peaks are shown in Table 3. This result has shown that the presence of quartz in the original ash gives rise in the IR spectrum to a series of bands located at 1268.24 and 798.56 cm⁻¹. The presence of mullite, in turn, is responsible for a series of bands at around 3024.48-3506.7cm⁻¹. The presence of carbon group is present in series of bands at around 4018.82-4555.05 cm⁻¹. Quartz, mullite

and the vitreous phase of the ash overlap in the area between 1220 cm^{-1} and 1434.12 cm^{-1} . Hence Quartz, Mullite, carbon and vitreous phases are confirmed to be present. This is in agreement with the earlier work of [4, 9].

The temperatures of destruction (T_{des}) of breadfruit seed hull ash, subjected to investigation, were determined from DTA curves. DTA data were recorded on “Derivatograph OD 102”, at heating rate of $10^\circ\text{C}/\text{min}$ in argon. The result of the TGA/DTA scan is show in Figure 7.

The pronounced endothermic effects of the observed in the DTA curves correspond to the oxidative degradation process and to the release of volatile matters (see Figure 7). On the TG curve a mass loss of 20% was observed in the temperature range of $700\text{-}900^\circ\text{C}$. DTG curve shows that the temperature of maximal decomposition/ destruction was 900°C (see Figure 7). Exhaustive investigation of thermal degradation of other biomass ash samples by mass spectroscopy performed by the authors [1, 3-4], demonstrated that in the temperature ranges of $750\text{-}800^\circ\text{C}$ which correspond to the first and second degradation step, the main volatile products were CO_2 and H_2O . The results obtained indicate that degradation processes of breadfruit seed hull ash was fully accomplished in the range $750\text{-}900^\circ\text{C}$.

4. Conclusions

From the analysis of the results and discussion given above, the following conclusions can be made.

- 1) XRD analysis of the breadfruit seed hull ash reveals : iron, Chromium Aluminum Silicon, Aluminum Chromium Iron, Potassium Sulfate, Sodium Magnesium silicate, Aluminum Silicon Carbide, while each of these phases have a score of 42, 30, 29, 34, 21,29 and 20 respectively.
- 2) XRF studies revealed the presence of hard element like SiO_2 , Al_2O_3 , MgO , Cr_2O_3 and Fe_2O_3 as major constituents which can be used as particulate reinforcements in MMCs for automobile applications.
- 3) FTIR graphs showed that Quartz, Mullite and Vitereous, carbon phases were present in breadfruit seed hull ash powder and proposed to use breadfruit seed hull ash as particulate reinforcement in MMCs.
- 4) The DTA/TGA result show that thermal stability of the breadfruit seed hull ash is up to 900°C
- 5) The breadfruit seed hull ash has a density of $1.98\text{g}/\text{cm}^3$ and can withstand a temperature of up to 1500°C . That means this ash can be use in production light weight MMCs component with good thermal resistance.

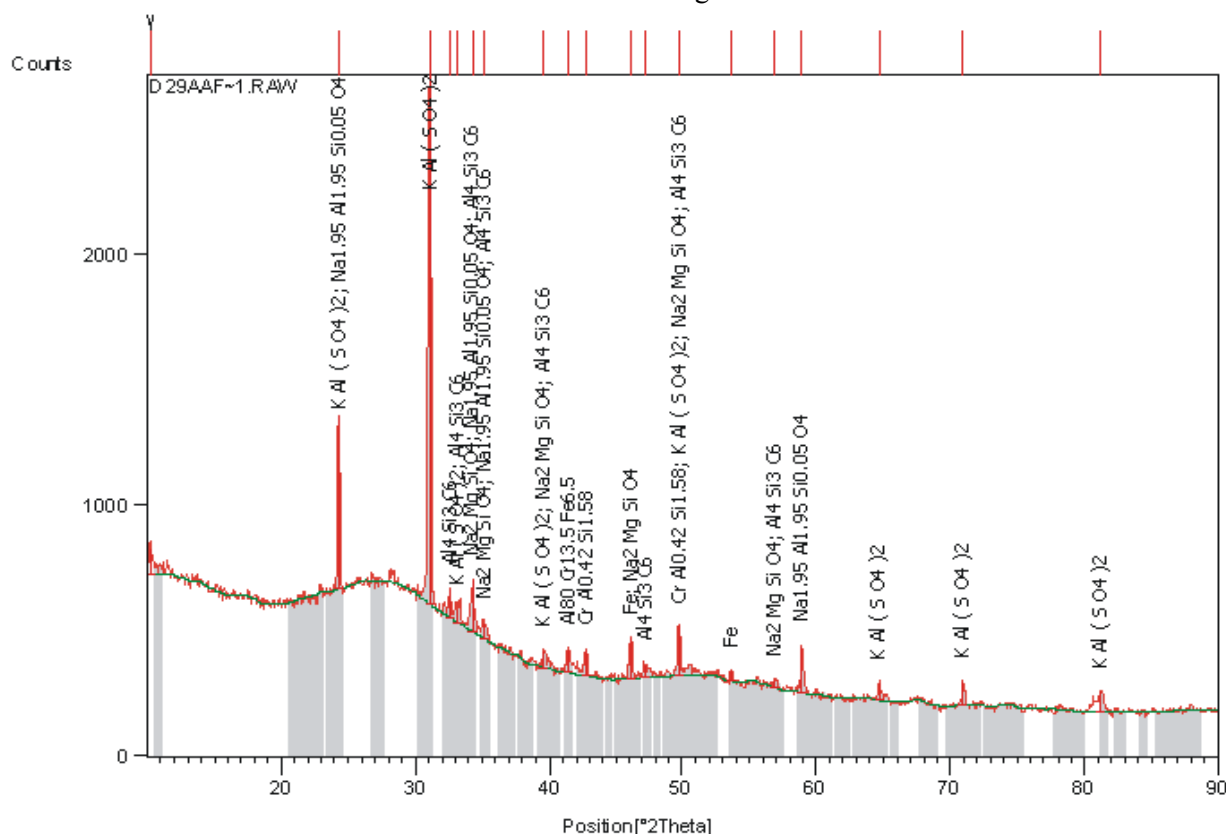


Fig. 3: XRD pattern of Breadfruit seed husk ash

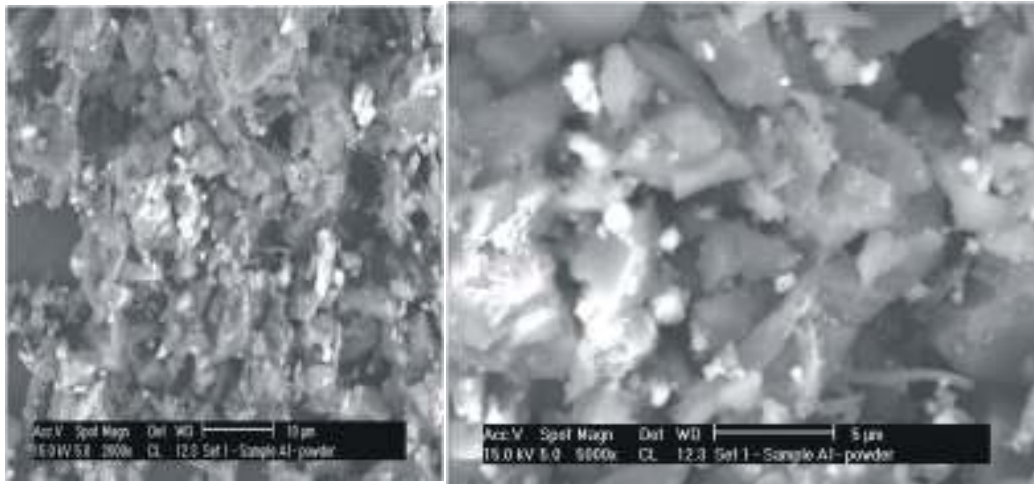


Fig. 4: SEM microstructure of breadfruit seed hull at 10 and 5micrometer

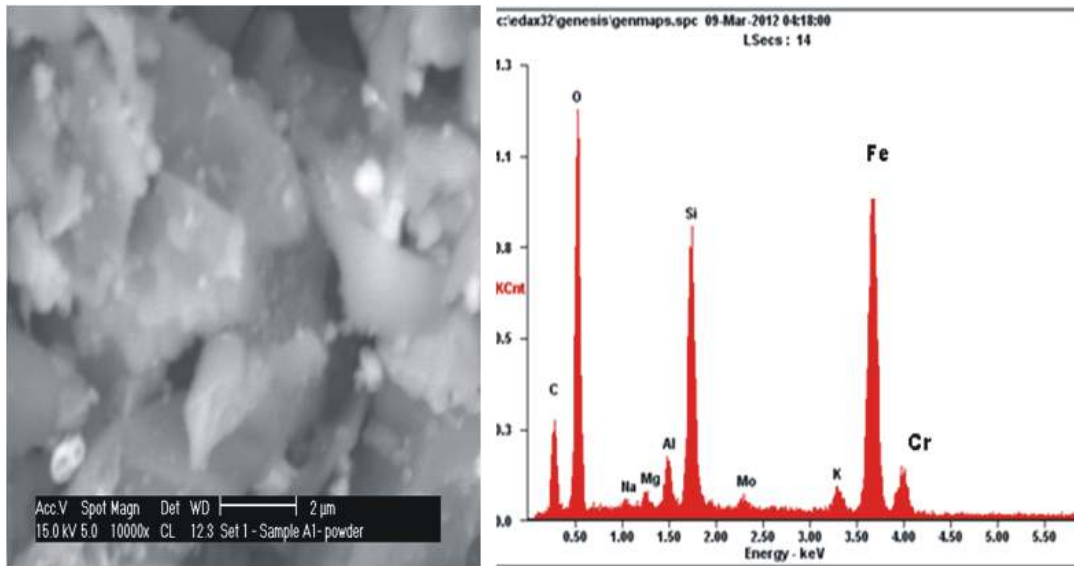


Fig. 5: SEM/EDS microstructure of breadfruit seed husk at 2micrometer

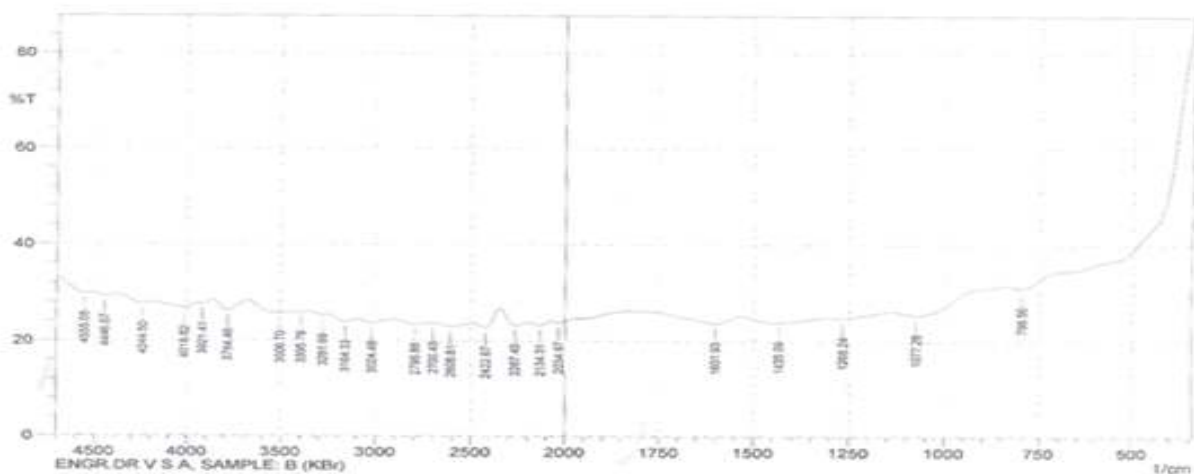


Fig. 6: FTIR spectrum of the breadfruit seed hull ash

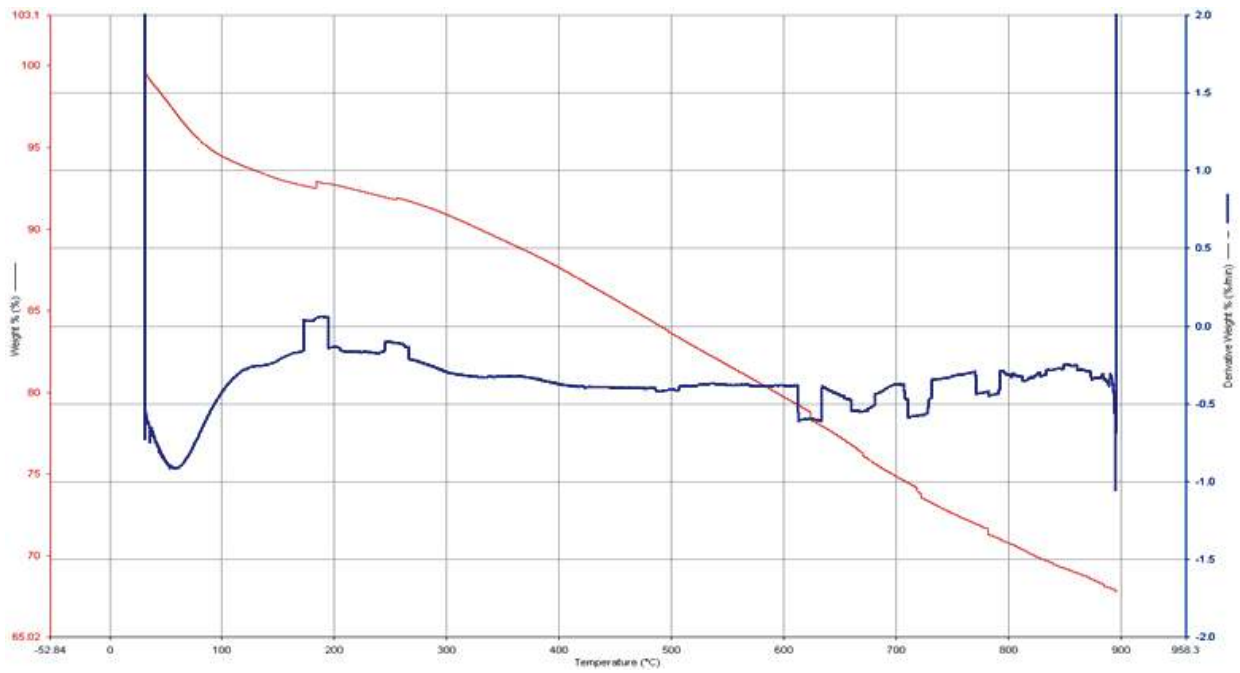


Fig. 7: DTA/TGA spectrum of breadfruit seed hull ash

Table 1: Identified Patterns List of Breadfruit seed husk ash

Visible	Ref. Code	Score	Compound Name	Displacement [°2Th.]	Scale Factor	Chemical Formula
*	01-088-2324	42	Iron	0.303	0.044	Fe
*	01-089-4866	30	Chromium Aluminum Silicon	0.051	0.037	Cr Al0.42 Si1.58
*	00-040-1243	29	Aluminum Chromium Iron	-0.134	0.035	Al80 Cr13.5 Fe6.5
*	01-070-2159	34	Potassium Aluminum Sulfate	-0.206	0.512	K Al (S O4) 2
*	00-034-1342	21	Sodium Magnesium Silicate	0.177	0.058	Na2 Mg Si O4
*	00-049-0003	29	Sodium Aluminum Silicate	0.426	0.078	Na1.95 Al1.95 Si0.05 O4
*	00-042-1172	20	Aluminum Silicon Carbide	0.127	0.032	Al4 Si3 C6

Table 2: XRF analysis of Breadfruit seed hull ash

Element	Al ₂ O ₃	Cr ₂ O ₃	Fe ₂ O ₃	K ₂ O	MgO	Na ₂ O	SiO ₂	MnO	ZnO
%	35.8	5.06	30.34	0.52	1.2	0.45	15.45	0.22	0.05

Table 3: FTIR Analysis of the breadfruit seed husk ash

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	798.56	31.16	5.281	843.88	339.48	204.541	60.52
2	1077.28	25.437	1.968	1139	843.88	162.415	3.8
3	1268.24	24.858	0.386	1302.96	1139.97	96.955	0.581
4	1435.09	23.931	1.17	1533.46	1303.92	140.469	2.602
5	1601.93	23.721	1.731	1826.65	1534.42	175.523	3.29
6	2034.97	23.796	0.67	2074.51	1827.61	149.048	1.235
7	2134.31	23.247	0.767	2193.14	2075.47	73.703	0.809
8	2267.4	23.246	2.041	2346.48	2194.1	93.952	2.98
9	2422.67	22.725	2.397	2486.33	2347.45	86.141	3.046
10	2608.81	22.967	0.728	2688.86	2487.29	127.397	1.384
11	2700.43	23.677	0.009	2733.22	2689.82	27.146	0.005
12	2796.88	23.432	0.518	2903.93	2734.19	105.858	0.758
13	3024.48	23.712	0.77	3096.82	2904.89	118.59	1.212
14	3164.33	23.954	0.982	3250.16	3097.78	93.136	1.358
15	3261.99	25.291	0.334	3346.61	3251.13	56.569	0.277
16	3395.79	25.783	0.194	3444.02	3347.57	56.621	0.162
17	3506.7	25.796	0.8	3671.62	3444.98	130.964	2.549
18	3784.46	26.228	2.234	3861.62	3672.59	106.592	3.402
19	3921.41	27.626	0.322	3946.49	3862.58	46.487	0.246
20	4018.82	26.788	1.01	4180.85	3947.45	131.542	1.983
21	4244.5	27.571	0.909	4388.2	4181.81	112.968	1.341
22	4446.07	29.227	0.551	4512.61	4389.17	65.422	0.5
23	4555.05	29.76	0.966	4700.68	4513.58	95.382	1.704

5. References

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