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ORIGINAL ARTICLE

Preliminary characterisation of the *Thaumatococcus daniellii* fruit as a potential biomass source for biorefinery

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Abstract
The inherent variability of biomass composition and structure, as well as the increased trends towards blending of biomass feedstock for energy, chemicals and materials, requires the systematic characterisation of biomass from both established and new sources. The high-value sweet protein thaumatin is produced from *Thaumatococcus daniellii* (*T.d*) fruit, native to West Africa, leaving about 90% of the fruit as waste. In this study, the proximate, ultimate, compositional and thermochemical characterisation of *T.d* fruit pulp and seeds, are determined, with a view to establishing the potential for a *T.d* biorefinery platform. Extractives content was determined through ethanol soxhlet extraction; cellulose, lignin and pectin contents by alkali hydrolysis, Klason method and acid hydrolysis respectively; while the thermochemical properties were determined by FTIR, EA, EDS and TGA. The proximate composition for pulp and seed biomass, respectively were; ash 17.47%/11.64%, moisture 16.29%/9.56%, fixed carbon 12.5/14.2%, extractives 15.7/3.4%, cellulose 25.34/26.82%, hemicellulose 21.61/15.89%, lignin 10.75/18.20%, pectin14.78/20.85%; elemental composition; C 34.27%/43.09%, H 4.7%/5.72%, N 1.08%/2.16% S 0.33%/0.15%, O 59.62%/48.88%. Analyses of the *Thaumatococcus daniellii* pulp and seed biomass samples revealed a relatively low lignin content, and high pectin and ash content. These characteristics indicate their potential as feedstock for low to medium value products such as pectin, cellulose and mineral-rich biochar. The potential for the recovery of a wide range of low to high value products from this biomass merits its consideration for a biorefinery platform. This should increase the economic value of this local plant while simultaneously alleviating pollution problems.

Keywords *Thaumatococcus daniellii* · Thaumatin · Fruit · Waste valorisation · Biorefinery concept

1 Introduction

The potential of biomass for different applications greatly depends on the characteristics of the biomass such as the chemical and structural composition, proximate and elemental content and thermal behaviour. Hence the need for systematic characterization of biomass components and their characteristics is particularly heightened in the context of the exploitation of biomass on integrated biorefinery platforms [1]. Biorefineries are facilities which use a range of technologies to process biomass feedstock to a wide range of bioproducts of diverse value within a single facility, in the same way petroleum-based refineries process crude oil for the production of diverse products. The principal products of biorefineries are platform chemicals and important economic products [2]. Biomass has also been combusted to produce energy [3, 4] and biomass waste has been converted into bio crude oils [5], ash and syngas [6]. Notable agricultural wastes that have been used in the biorefinery sector to produce value-added products include waste from the processing of wheat and corn [7, 8]: sugarcane bagasse [9] and rice straw [10].

Agro-industrial wastes are a sustainable source of lignocellulosic biomass, especially because they do not compete with food supply and their usage serves as a way of eliminating waste which would otherwise be a source of pollution

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[11]. The composition of lignocellulosic biomass gives it great potential for transformation into valuable products and chemicals within biorefineries [12–14].

Thaumatococcus daniellii (denoted as *T.d*) is a herbaceous plant of the Marantaceae family, native to West and Central Africa. This plant is the source of a high value sweet protein, Thaumatin (a non-calorific sweetener, about 3000 times sweeter than sucrose) which is extracted from arils of the *T.d* fruits. This aril is reported to be just about 4.8% (w/w) of the entire *T.d* fruit, while 95.2%, made up of the seeds and pulp, is left as waste [15]. The fruits are typically foraged from forests, which are their natural habitat. The literature reports on this plant mainly stem from its local applications such as; the leaves, used for wrapping and cooking food, the fruit, for sweetening sour foods and fermented wine [16] the leaves and stems for building local huts [15, 17] and in traditional medicine as an antidote against venoms, stings and bites and the seed as a local emetic, for treating pulmonary problems [18]. Different parts of the plant have been found to display good nutritional quality with essential oils of the fruit tissue and seed containing significant quantities of potassium, calcium, sodium, magnesium, and oxalate [19]. Extracts from different parts of the plant have been shown to have interesting antioxidant and antimicrobial activities and to have a rich phytochemical composition, significant quantities of flavonoids, alkaloids and tannins [15, 18, 20]. Numerous studies on the leaves have reported on the phytochemistry and anti-microbial evaluation of *T.d* leave extracts, revealing antimicrobial activity against food-spoilage bacteria and yeasts, as well as an overview of the plant's traditional uses for flavoring, packaging, roofing, weaving and as a local remedy [21, 22]. In addition, chemical evaluation of waste from thaumatin production reported interesting proximate and nutrient content with potential for use in feed formulations [23]. Numerous research efforts have been conducted on *T.d* as an economic plant while highlighting its versatile uses in several West and Central African countries, including Nigeria [24, 25]; Ghana [18]; Ivory Coast [22, 25] and Cameroon [16, 17, 26]. However, limited information exists on the proximate and ultimate composition of the biomass alongside their compositional, thermal and chemical characterization, for potential application of the biorefinery concept.

Thaumatococcus daniellii, locally known as Ngongo leaf in some areas of Cameroon [17], Katemfe in Ghana, and Sweet Prayer Plant and/or Miracle berry in Nigeria [27], is naturally, found growing under cover, especially in cocoa plantations, making them suitable for co-cultivation [17, 27]. Comparatively, thaumatin appears to be the sweetest substance known, with applications as a sweetener, flavour enhancer, food additive E957, as well as in confectionaries, beverages and in the drug industry [28, 29]. The non-calorific sweetening property of thaumatin makes it an ideal sweetener for patients with diabetes and a healthier alternative to calorific sweeteners. For this reason, its demand is on the rise, and projections on market demands are expected be at USD 70 million with a compound annual growth rate of 6.1% by 2027 [29]. With the large amounts of waste generated from thaumatin production, projections on the summative polluting effect indicate that it could be highly environmentally and economically unfriendly in the nearest future, if left unattended [24, 30]. We report here, the thermochemical analysis, proximate, and ultimate composition of the seed and pulp biomass, as well as the compositional analysis, to determine the lignin, cellulose, hemicellulose, pectin and ash content of the seeds and pulp of the *T.d* fruit in an attempt to demonstrate the feasibility of a *T.d* fruit based biorefinery.

2 Methodology

2.1 Sample collection and preparation

Mature ripe *Thaumatococcus daniellii* fruits were harvested from local farms in Bulu Native, Buea, South West Region of Cameroon (4°08′25″ N 9°17′33″ E), and transported to the Inorganic Laboratory of the Department of Chemistry, University of Buea. The fresh fruits were washed and dried using a cotton cloth. Manual separation of the fruit parts was performed: the fruit pulp was separated from the seed, and the seed was separated from the aril (a layer of gelatinous material found on the seed, which is used for the manufacture of thaumatin). The fruit pulp was cut into smaller sections and together with the seeds were oven dried at 50 °C over a period of 24 h, cooled to ambient temperature, then, ground using a mechanical mill. The powder was sieved through a 300 µm sieve and stored in vacuum for characterisation.

2.2 Moisture content

2.0 g of each sample was weighed using a chemical balance and subjected to heating in an oven at 105 °C for 6 h after which they were removed and cooled to room temperature (25 °C) in a desiccator and the mass recorded[31]. The amount of water evaporated from the sample was designated as the moisture content and it was obtained using Eq. (1). The measurements for each sample were done in triplicates and the average values recorded.

$$Moisture\ content\ (MC\ \%) = \frac{Wa - Wb}{(Wa)} \times 100 \quad (1)$$

W_a weight of the raw sample
W_b weight of water-free sample

2.3 Ash content

Ash content was determined in triplicate and the final results were recorded as the mean. The dried biomass samples were combusted in a muffle furnace at 550 °C for a period of 5 h [32, 33]. The residue was cooled and weighed and the ash content was expressed as a percentage in Eq. 2.

$$Ash\ content\ (AC\ \%) = \frac{Weight\ of\ biomass\ sample(Wa) - weight\ of\ incinerated\ sample(Wb)}{Weight\ of\ biomass\ sample(Wa)} \times 100 \quad (2)$$

2.4 Volatile matter content

1.0 g each of dry pulp and seed samples were weighed in a ceramic booth with lid covered and placed in a furnace preheated to 910 °C for 7 min. At the end of this process, the samples were cooled, weighed, and stored in a desiccator [34]. The amount of volatile matter in the biomass was expressed as a percentage difference in weight, Eq. 3. The measurements for the procedure were done in triplicates and the mean was recorded.

$$Volatile\ Matter(\%) = \frac{W1 - W2}{W1} \times 100 - MC(\%) \quad (3)$$

where.

W1 weight of the sample before heating,
W2 weight of the sample after heating
MC moisture content

2.5 Fixed carbon

The fixed carbon content of the biomass samples was obtained by subtracting the percentages of Moisture Content, Volatile Matter, and Ash Content from 100 [35], Eq. 4.

$$Fixed\ Carbon(\%) = 100 - moisture\ content(\%) - ash\ content(\%) - volatile\ matter(\%) \quad (4)$$

2.6 Total solids

The total solid content was obtained by subtracting the percentage by mass of Moisture from 100 [35], Eq. 5.

$$Total\ solids(\%) = 100 - MC(\%) \quad (5)$$

2.7 Ultimate analysis

The elemental content (carbon, hydrogen, nitrogen and Sulphur) of the samples were determined using a Thermo Fisher Scientific FLAS 2000 CHNS/O Elemental Analyzer Instrument with BBOT (2, 5-Bis (5-terbutyl-bezoxazole-2yl) thiophene) as the standard to obtain mass percent composition of C, H, N, and S. The oxygen composition by was obtained by difference, Eq. 6.

$$Oxygen\ content(\%) = 100 - (C\% + H\% + N\% + S\%) \quad (6)$$

2.8 Higher heating value (HHV)

HHV is the total amount of heat energy in a fuel, including the energy in the water vapour of the exhaust gases. The HHV of biomass samples are useful for examining the potential of biomass to produce biofuel. These values in lignocellulosic biomass are dependent on soil composition, minerals, and weather conditions where the biomass is found. The value was obtained using Eq. 7, stipulated by the International Energy Agency (IEA) [35].

$$HHV(MJ/kg) = 0.3491 \times C(\%) + 1.1783xH(\%) + 0.1005 \times S(\%) - 0.0151 \times N(\%) - 0.1034 \times O(\%) - 0.0211 \times AC(\%) \quad (7)$$

where.

C carbon), H (Hydrogen
S Sulphur
N Nitrogen
O Oxygen
AC Ash content

2.9 Removal of extractives

The extractives were removed from the samples by Soxhlet extraction for 8 h using distilled ethanol as extractant (standard: ASTM E 1690 – 01) [36]. The samples were dried in an oven at 50 °C till a constant weight was achieved, and the samples were referred to as extractive free samples (EFS), used to test for compositional components such as hemicellulose, cellulose and lignin.

2.10 Characterisation of extractives-free biomass

2.10.1 Holocellulose content

To determine the holocellulose (α-cellulose + hemicellulose) content, the extractive free samples were treated with 0.7 w/v.% sodium hypochlorite, buffered at pH 4 and refluxed for 90 min at 90–95 °C using a sample-to-liquid ratio of 1:50. 5% w/v sodium metabisulphite solution was added, and allowed for 15 min to stop the chlorite action, and the sample was then filtered and washed several times with distilled water. A cheesecloth was used to squeeze out excess water and the sample was dried in an oven at 50 °C till constant weight [37, 38].

2.10.2 Cellulose and hemicellulose content

The dried holocellulose samples were treated with 20% NaOH solution for 4 h with a sample-to-liquid ratio of 1:100, with occasional stirring. The hemicellulose dissolved into solution and α-cellulose was the residue. The suspension was filtered and the residue washed several times with 2% acetic acid and distilled water, and squeezed to remove excess water using a cheesecloth and dried in an oven at 50 °C to a constant mass. The α-cellulose content was obtained by weight difference between holocellulose and the hemicelluloses [37].

2.10.3 Lignin content

The extractive free samples were treated with 72% (v/v) sulphuric acid with a 1:15 w/v ratio, with regular stirring in an ice bath at 4 °C for 5 min (Klason method). The samples were then kept for 2 h at room temperature with regular stirring. The suspension was transferred to a 1000-ml flask, and 345 mL of distilled water added to reduce the acid concentration to 3%, followed by refluxing for 4 h. The suspension was filtered, using a Buchner funnel with a whatman n^{o} 1 filter paper, washed several times with hot distilled water and dried at 50 °C till constant weight, and the weight recorded [37].

2.10.4 Pectin isolation

Pectin was isolated in aqueous medium, under reflux at 97 °C for 30 min, at pH of 2 using HCl. The suspension obtained was filtered through a cheesecloth and the filtrate cooled to room temperature and treated with excess 95% ethanol, this yielded a precipitate. This was filtered using a cheesecloth and dried at 50 °C till constant weight [38].

2.11 Characterisation

The FT-IR spectrum of each biomass samples was recorded over the range 4000–400 cm^{-1} using a PerkinElmer FT-IR GX instrument. 200 mg of dried potassium bromide was used to infuse 2 mg of the biomass sample, and the mixture was compressed to pellets. A TGA Q500 V20.10 Build 36 Universal V4.5A TA instrument was used to carry out the Thermogravimetric measurements, over the temperature range of 20 to 1000 °C with a heating rate of 20 °C/min. The measurements were done under a nitrogen atmosphere to prevent thermoxidative degradation. Throughout the process, data of the sample weight was recorded as a function of time and temperature and displayed on a derivative curve (DTG). The mineral composition of the biomass ash was obtained using a Zeiss Gemini Ultra Plus FEG scanning electron microscope coupled to an energy dispersive X-ray Spectrometer (EDX). Sample preparation was done by coating samples with Au/C using a vacuum sputter, after which they were placed in the sample holder for analysis.

3 Results and discussion

3.1 Physical characterisation of the fruit

The whole fruits (A) and cross section (B), fruit pulp or pericarp (C), seed (C), and aril (D), attached to the seed are presented in Fig. 1, while Table 1 shows the physical parameters of the fruit parts.

The average matured fruits contained a total of three seeds and weighed approximately 24.48 g each. The percent by mass of each of these parts was evaluated for fresh fruits, and the fruit pulp, seed, and aril were found to constitute 69.85, 19.41 and 10.47% of the total mass of the fruit, respectively. These results indicate that 89.26% of the fruit is left to waste during the extraction of the sweet protein Thaumatin. The values reported in this study are comparable with those reported in literature (72.4, 22.8 and 4.8% for pulp, seed and arils respectively) [15]. The results validate previous reports that over 90% of the fruit is left as waste in the environment during the extraction of Thaumatin from the fruit.

3.2 Proximate analysis

A comparison of the proximate composition of the *T.d.* fruit pulp and seeds is represented in Table 2.

The dried biomass sample ash contents of the pulp (17.47%) and seed (11.64%) were within the range reported in literature (5.8–21.08%) and (8.17–11.64%) for pulp and seed, respectively [15, 23, 24, 39]. The moisture content of

Fig. 1 Parts of the *Thaumatococcus daniellii* fruit; Whole fruit (**A**), Pulp and Seed (**B** and **C**) Aril (**D**)

Table 1 Physical parameters of the fruit

Part	Parameter	Average value	% of total weight	Total %
Whole fruit	Total fruit weight	24.58 g	100	100
	Length	3.97 cm		
	Width	3.27 cm		
	Number of seeds	3		
Fruit pulp	Total weight	17.17 g	69.85	**89.26**
Seed	Total weight of all seeds without arils per fruit	4.77 g	19.41	
Aril	Total weight of arils on all seeds per fruit	2.64 g	10.74	10.74

the pulp (16.29%) was within the range reported in literature for *T.d.* pulp (10.04–89.2%). The difference in values was attributed to the state of the biomass samples used for the moisture content determinations; the high values (83.9 and 89.2%) observed in literature were reported on biomass samples on a wet basis [23, 39] whereas, the determinations in the present study were done on biomass samples on a dry basis which recorded similar values (10.04%) to a study which was done on dry biomass samples [15], (Table 2). The moisture content of the pulp was equally less than 48.0–50.2% reported for sugarcane bagasse (Table 3). On the other hand, the moisture content of the seed (9.56%) is slightly below the range reported in literature for *T.d.* seeds (10.39- 31.80%) (Table 2). The relatively low moisture

Table 2 Comparison of proximate composition of *T.d.* pulp and seed

Part	Ash content (%)	Moisture content (%)	Volatile matter (%)	Fixed carbon (%)	Total solid (%)	Literature
Pulp	5.80	83.9	-	-	-	[39]
Pulp	21.08	10.04	-	-	-	[15]
Pulp	20.70	89.20	-	-	-	[23]
Pulp	17.47	16.29	53.72	12.52	83.71	Present study
Seed	8.17	10.39	-	-	-	[19]
Seed	11.30	15.15	-	-	-	[15]
Seed	9.18	10.52	-	-	-	[24]
Seed	9.10	31.80	-	-	-	[23]
Seed	11.64	9.56	64.62	14.18	90.44	Present study

Table 3 Detailed characterisation of *T.d.* pulp and seed compared with other reported biomasses

Proximate analysis											
Biomass	*T.d.* pulp	*T.d* seed	Corn stover [49]	Wheat straw [46, 50]	Sugarcane bagasse [9, 35]	Rice husks [51, 52]	Rice straw [53]	Linseed [54]	Amaltas seeds [35]	*Samanae saman* [55]	Sapodilla [56]
AC (%)	17.47	11.64	10.5	11.2–16.09	1.6–2.9	12.5–16.1	7.8–20.3	10.50	6.30	3.06	1.19
MC (%)	16.29	9.56	15	15	48.0–50.2	8.0–12.67	10.80	6.53	6.03	6.19	8.07
VM (%)	53.72	64.62	78.1	64.42–83.9	84.2	68.20–71.24	35.0–72.4	76.95	79.65	76.00	77.02
FC (%)	12.52	14.18	11.2	19.94	11.8	15.7–16.27	11.3–16.1	6.02	8.06	14.74	13.73
TS (%)	83.71	90.44	85	85	49.8	92	89.2	93.47	93.74	93.84	91.93
VM/FC	4.37	4.56	6.96	-	7.14	-	-	12.78	9.82	5.156	5.60
Ultimate analysis											
C (%)	34.27	43.09	44.5–46.7	37.2–43.9	44.6–49.2	35.86–45.2	36.0–41.0	61.00	42.37	48.46	52.67
H (%)	4.70	5.72	5.49	5.26–5.57	4.7–6.2	4.40–5.8	2.54–5.5	8.50	7.51	6.75	6.74
N (%)	1.08	2.16	0.67–0.8	0.63–1.14	0.18–0.5	0.28–1.02	0.6–4.43	3.00	2.98	7.30	1.46
S (%)	0.33	0.15	0.1	0.16–0.20	0.02–0.1	0.21	0.1–0.53	0.30	0.39	-	0.34
O (%)	59.62	48.88	38.4–39.5	37.3–38.7	43.0	47.6–59.46	36.3–52.61	27.2	46.74	37.47	38.78
O/C	1.74	1.134	0.8222	0.881	0.964	-	-	0.004	0.83	0.58	0.55
H/C	0.127	0.133	0.117	0.120	0.139	-	-	1.66	2.11	1.67	1.54
C/N	31.73	19.95	69.70	69.68	89.2	-	-	-	16.56	-	-
CHO index	3.34	2.14	-	-	-	-	-	-	1.85	1.26	1.21
HHV(MJ/kg)	10.54	16.47	13.6–18.5	14.56–17.5	9.8–17.4	15.18–16.35	-	27.9	18.68	17.68	20.05
Bulk Density (kg/m^3)	518	569	-	-	-	-	-	-	585.20	657.41	454.30
Compositional analysis of the study compared to typical lignocellulosic biomass [35, 50–53, 57–59]											
	T.d. pulp	*T.d.* seed	Corn stalk	Wheat straw	Sugarcane bagasse	Rice husk	Rice straw	Orange peels	Apple pulp	General range	
Extractives (%) (EtOH)	15.70	3.40	4.78	12.9	-	2.32	-	-	-	2.3–15.7	
Cellulose (%)	25.34	26.82	35.0–39.6	35.0–39.0	25.0–45.0	28.7–35.6	29.2–34.7	-	-	25.0–45.0	
Hemicellulose (%)	21.61	15.89	16.8–35.0	23.0–30.0	28.0–32.0	12.0–29.3	23.0–25.9	-	-	12.0–35.0	
Lignin (%)	10.75	18.20	7.0–18.4	12.0–16.0	15.0–25.0	15.4–20.0	17.0–19.0	-	-	7.0–25.0	
Pectin (%)	14.80	20.90	-	-	-	-	-	20–35	4–19	4.0–35.0	

AC, ash content; *MC*, moisture content; *VM*, volatile matter; *FC*, fixed carbon; *TS*, total solids; *HHV*, higher heating value

content of the T.d. seeds suggests that the biomass can be handled with ease (in terms of storage and transportation) [40], and indicates a high heating value for the biomass sampled in this study [41]. This also ensures the quality of pyrolysis products such as bio-oil and the quality of the volatile matter, rendering the material more suitable for the production of biofuels. On the other hand, the fruit pulp samples with relatively high moisture content may be less suitable for the production of biofuels but can be exploited in the production of biomaterials. The ash and moisture contents of the sampled biomasses were associated with eco-geography of the plant (the soil, climate, and geometry). Therefore, the difference between the literature values and the results of the present study can be attributed to the difference in the eco-geography of the plants. However, the results are in line with literature values obtained for conventional biomass already in use for biorefinery input chains: moisture content in the range of 8 – 15% reported for corn stover, wheat straw, rice husk and rice straw, and ash contents in the range of 1.19—20.3% reported for sugarcane bagasse, corn stover, wheat straw, rice husk and rice straw. Also for seed sample moisture content of 3.0—7.0%, pulp moisture contents of 6.26—6.55%, and ash content of 4.63—5.603% for non-edible oil seeds of putrannjiva, amaltas and siris plants, endemic to forests across tropical and subtropical countries [35, 42]**.** The volatile matter of biomass, which is the condensable vapor and permanent gases (excluding water vapour) liberated from biomass upon heating, recorded values of 53.72% and 64.62% for the pulp and seed samples, respectively. These values are very significant and suggest good ignition ability of the samples [43], indicating that the biomass under study, especially the seed has potential for use in the production of energy through combustion.

Ash content represents the amount of the solid residue remaining after the complete burning of the biomass sample; a high ash content implies a low level of volatile matter in a given material or fuel. The seed biomass in the present study had a lower ash content than the pulp biomass, and moderate volatile matter content suggesting that the seed biomass could be more suitable than the pulp for use as a fuel but even more preferably as an additive in bio composites [44, 45], as an ash content of 11.6—17.5% falls within the range reported for the conventional biomasses used in the biorefinery industry (Tables 2 and 3). From Table 3, it can be observed when comparing the ash contents of the reported biomass samples that the ash contents of the *T.d* pulp and seed biomass in the present study are relatively high, especially that of the *T.d* pulp which exceeded the range presented for wheat straw and rice husk. High ash content in biomass has been found to be disadvantageous to thermal processing routes like pyrolysis and gasification of biomass for the generation of bioenergy. Hence, high-ash biomass samples are usually used for low-value products. However, continuous research has led to the discovery of a new strategy where high-ash biomass was used for the production of biocomposites for large-scale 3D printing applications that possess adequate mechanical properties with potential to create new, high-value streams for the high-ash biomass [46]. The biomass in that study had ash contents in the range 0.7–11.9% [46], which corroborates with the ash contents of the *T.d* seed biomass in the present study (11.6%) and the *T.d* pulp (17.5%),which exceeds the highest value in the reported range, suggesting the possibility of better usage for biocomposites applications.

Fixed carbon is the solid combustible residue left after removing volatile matter by heating biomass. In this study, the highest value of fixed carbon was observed in seed samples, corresponding to the highest volatile matter content, and similar observations were made in literature [43]. Furthermore, the values in the present study (12.5—14.2%), fall within the range reported in literature (6.02—19.94%) (Table 3) and confirms the fact that the biomass is suitable for the production of biofuels. Results from proximate analysis of the biomass samples in this study suggest that they are of significant calorific value and would be suitable for biofuels as well as biomaterials.

3.3 Ultimate analysis

The elemental analysis results of the samples alongside the literature values obtained for other seeds and biomass samples are presented in Table 3. The results revealed a higher carbon content in the seed (43.09%), corresponding to a higher oxygen content, 48.88%, which resulted in a heating value of 16.47 MJ/kg for the seed which falls within the range for conventional biomass used in the biorefinery industry reported in the literature (9.2–18.5 MJ/kg), (Table 3). Hydrogen found in the range 4.7—5.72% falls within the range reported in literature (2.54—5.8%) for the conventional biomass used in the biorefinery sector. The percentage composition by mass of other elements such as nitrogen and sulphur was in the range 1.08—2.216% and 0.150—0.33% for the pulp and seed, respectively and fall within the range 0.18—4.43% for nitrogen and 0.02—0.53% for sulphur reported in literature (Table 3).The low value of nitrogen and sulphur correspond to low NO_x and SO_x emissions during pyrolysis, which consequently has the potential to reduce corrosion problems in boilers and pipelines [21]. The oxygen content fell within the range (48.88—59.62%) and given the variability in the eco-geography, compared well with the literature range (35.86—49.2%) (Table 3). Therefore, with the help of these findings, favorable catalysts for the removal of oxygen and heteroatoms from bio-oil derivatives can be identified to aid the pyrolysis of sample seeds. The C/N ratio influences the decomposition rate (higher C/N ratio leads to a decreased decomposition rate);

in the present study, pulp samples had relatively higher C/N ratio (31.73) relative to 19.93 for seeds, which is a value that is comparable to16.56 from literature (Table 3). The results revealed a low C/N ratio for the samples under study (compared to the values for some conventional biomass samples), indicating that the samples in this study may be more suitable for biogas production as their low C/N ratio favors decomposition by microorganisms [47].

3.4 Higher heating value and bulk density

The higher heating value was a result of a combination of proximate and elemental analyses of the biomass materials. Higher heating values are related to water retention in the combustion product. Higher heating values in this study: 10.5 MJ/kg for pulp and 16.47 MJ/kg for seed fall within the range reported for conventional biomass such as corn stover, wheat straw, sugarcane bagasse, rice husks and rice straw used in the biorefinery industry (9.8—18.7 MJ/kg) (Table 3). The values are attributed to the amount of carbon, hydrogen, oxygen, and ash. However, these amounts vary with the geographical location of the plant and the harvest season. Therefore, the range for comparison is sufficiently large and accommodates the values obtained in this study. The bulk density obtained in the range 518—569 kg/m^3, also falls within the range 454.30—657.41 kg/m^3 (Table 3) reported in literature for the other conventional biomasses used in the biorefinery sector. These higher bulk densities confirm that the samples can be handled with ease.

3.5 CHO index

The CHO index is directly proportional to the number of molecules of oxidized compounds but inversely proportional to the heating value of a biomass (lowest CHO index indicates reduced number of molecules of oxidised compounds and high CHO index indicates high number of molecules of oxidised compounds). A high CHO index indicates a low heating value. The CHO index observed in this study were 3.34 for pulp and 2.14 for seed, indicating that the biomass from seeds had a higher heating value and lower number of molecules of oxidized compounds relative to pulp. Furthermore, the CHO index for the seed samples was closer to literature values (1.85) for amaltas oil seed biomass (Table 3), suggesting that the biomass from the seed samples in the present study could behave similarly during catalytic pyrolysis [35].

3.6 H/C ratio

The molar carbon to hydrogen ratio in biomass, has been found to be an indicator of the materials degree of carbonisation and stability and hence its suitability for applications such as in the production of biochars [48]. Biochars are a porous carbonaceous solid material formed by the thermal decomposition of biomass, with applications as low-cost catalysts, in the treatment of wastewater, for composting and for soil enhancement. It has been established that biomass H/C ratios greater than 0.7 are an indicator for subsequent low-quality biochar. This is because, pyrolysis causes the devolatilsation of biomass and the carbon enrichment of the solid fraction. For biomass with H/C ratios greater than 0.7, hydrogen and oxygen are preferentially absorbed over carbon, and the H/C and O/C ratios have a tendency to decrease when biomass conversion into biochar occurs [48]. This suggests that the *T.d.* pulp and seed biomass in the present study could be very much suitable for the production of biochars as the H/C ratios were 0.127 and 0.133 for the pulp and seed biomass respectively, far less than the 0.7 mark for low quality biochars.

3.7 Compositional analysis

The pulp and seed sample biomasses analyzed have a significant quantity of fermentable sugars in their composition (i.e. holocellulose which includes hemicellulose and cellulose), also referred to as total structural carbohydrates, with total percentages of 46.95 and 42.71% for pulp and seed, respectively, which are greater than the minimum 40% of holocellulose necessary for input in the supply of bioproducts such as platform chemicals and biochar and platform chemicals. This can be seen in Table 3 where the holocellulose content of rice husk ranges from 40.4–64.9 of which the values of holocellulose for T.d pulp and seed fall within this range [51, 52] [25]. Lignocellulosic biomass with structural carbohydrate contents exceeding 40% has also been demonstrated to be feasible for fermentative and enzymatic processes [60]. This indicates that the pulp and seed biomass in the present study where the structural carbohydrate contents were 46.95 and 42.71% respectively, could be suitable for bioconversion processes. As a matter of fact, the cellulose composition of the T.d pulp and seed fall within the cellulose composition range for sugarcane bagasse (Table 3), which is one of the most prominent and widely used biorefinery feedstock globally [9, 35] Therefore, these samples are good candidates for input into bioproduct supply chains. The hemicellulose content of the T.d pulp and seed falls within the range for corn stalk and rice husk (Table 3). The extractive values (15.7 and 3.4% for sampled pulp and seed biomasses respectively), are moderate to low and may not necessitate removal in the input chain [61], making the process more economical… The levels of lignin in both biomass samples from pulp (10.75%) and seed (18.20%) are relatively low but have potential for acid hydrolysis processes [56, 62]. These values are however close to reported

values for corn stalk, wheat straw, sugarcane bagasse, rice husk and rice straw (Table 3) which are among the major biomass feedstock used for biorefinery globally. High lignin content has been found to decrease the efficiency of enzymatic hydrolysis of cellulose as bonding occurs between the enzyme and lignin, which then blocks the active sites. Hence, the relatively low lignin content of the *T.d* seed and pulp biomass in the present study again suggests that this biomass may be suitable for bioconversion processes such as anaerobic fermentation to obtain platform chemicals such as short chain volatile fatty acids, which can be processed chemically by hydrogenation and esterification to obtain mixed alcohols that include ethanol, propanol and butanol, which can be used as biofuels or as platform chemicals [63].

The pectin content of 14.8 and 20.9%, respectively for the *T.d* pulp and seed biomass in the present study falls below the range of pectin extracted from citrus peels (containing 20–35% pectin) which is one of the richest sources of commercial pectin, but comparable to pectin content of apple pulp/pomace (containing 4–19%) which is also a major source of commercial pectin. Pectin is rich in neutral sugars and has applications ranging from the food industry where it is used as, thickening, stabilizing and a gelling agent, to the pharmaceutical sector where it is used in drug delivery, tissue engineering, and wound healing, and as source of oligomers used in the nutraceutical industry as probiotics [57–59]. The close relationship between the pectin content of the T.d Pulp and especially the T.d seed to the pectin content of apple pomace and citrus peels, illustrates the potential of the T.d fruit for pectin extraction and direct valorisation of the biomass.

3.8 Fourier transform infrared spectroscopy of the biomass

The FTIR analysis presented in Fig. 2 shows the functional group characterization of pulp and seed samples. The IR profiles of all the samples were similar in terms of the number of bands and band frequencies, indicating that the composition of the samples from the parts of the *T.d.* plant samples are similar, particularly with respect to their structural content [49, 50]. The profile shows multiple peaks indicating the presence of many functional groups such as alkene, carbonyl and aromatic rings. The band within the range 3300—3315 cm^{-1} indicates the presence of protein, phenols, alcohols and water of absorption characteristic hydroxyl group, stretching vibrations [16]. The band located at 2910–2930 cm^{-1} is characteristic of the aliphatic C–H stretching and signals the presence of fermentable sugars such as hemicellulose and cellulose [42]. The peak at 2929 cm^{-1} is more intense in the seed sample can be assigned to O–CH_3, vibration characteristic of lignin. The absence of the peak at 1730—1750 cm^{-1} associated with carbonyl groups (C = O) indicates the absence or low concentration of p-coumaric acids in lignin and acetyl group in hemicellulose. The peak in the range 1585 – 1600 cm^{-1} is characteristic of C = O asymmetric stretching vibrations of C = O of the free carboxyl in polygalacturonic acid in pectin. The peak that profiles in the range 1540–1650 cm^{-1} also indicates the occurrence of C = C in aromatic ring stretching and the N–H amines groups. The band in the range 1400–1450 cm^{-1} is attributed to the aromatic methyl group. This band indicates that cellulose and hemicellulose are present. The bands at 1222 cm^{-1} are ascribed to the aryl group in lignin while the peaks at about 1174 cm^{-1} and 1359 cm^{-1} are attributed to C–O–C stretching and –CH_2 wagging, respectively, usually found in cellulose and hemicellulose networks. The band at 1040 cm^{-1} is assigned to the stretching vibrations of C–O usually observed in polysaccharides and lignin. The intense peak in the region 450–800 cm^{-1} assigned to the C–H out of plane twisting indicates that sugars such as galactan, B-D fructose, and arabinan are present [64].

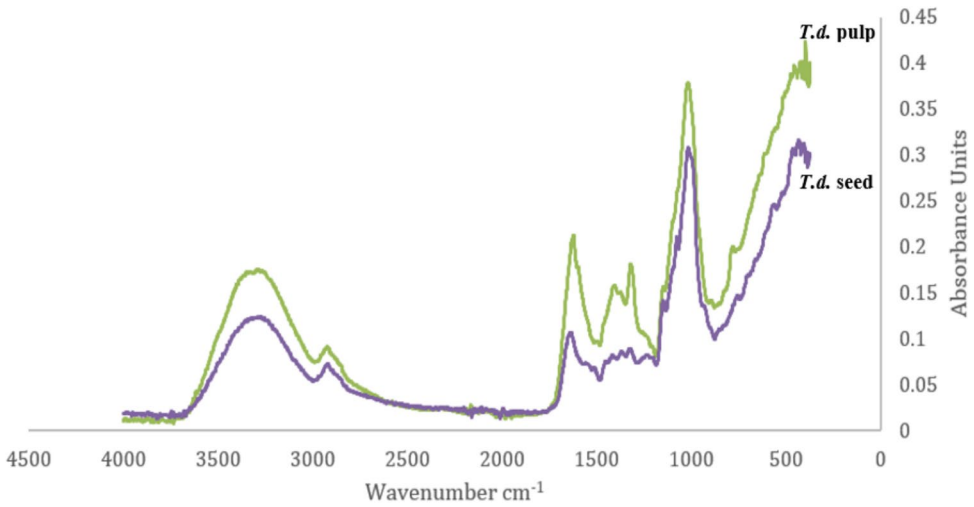
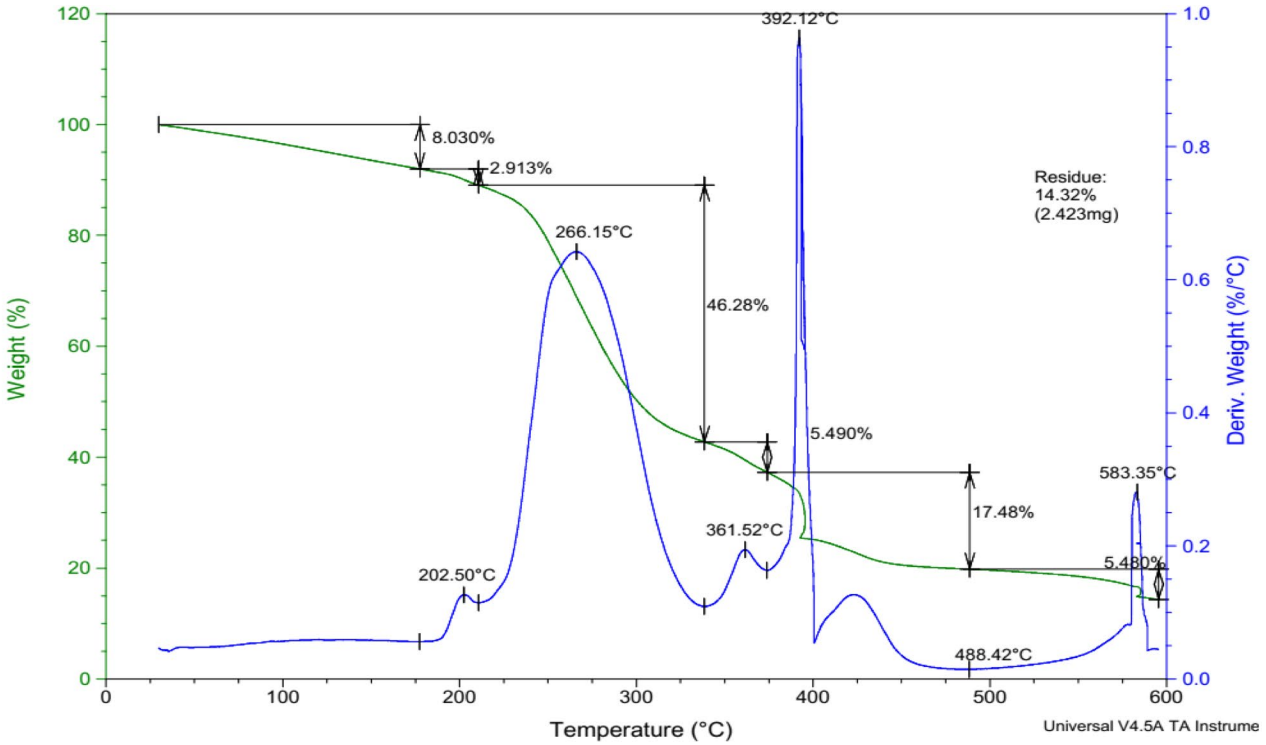
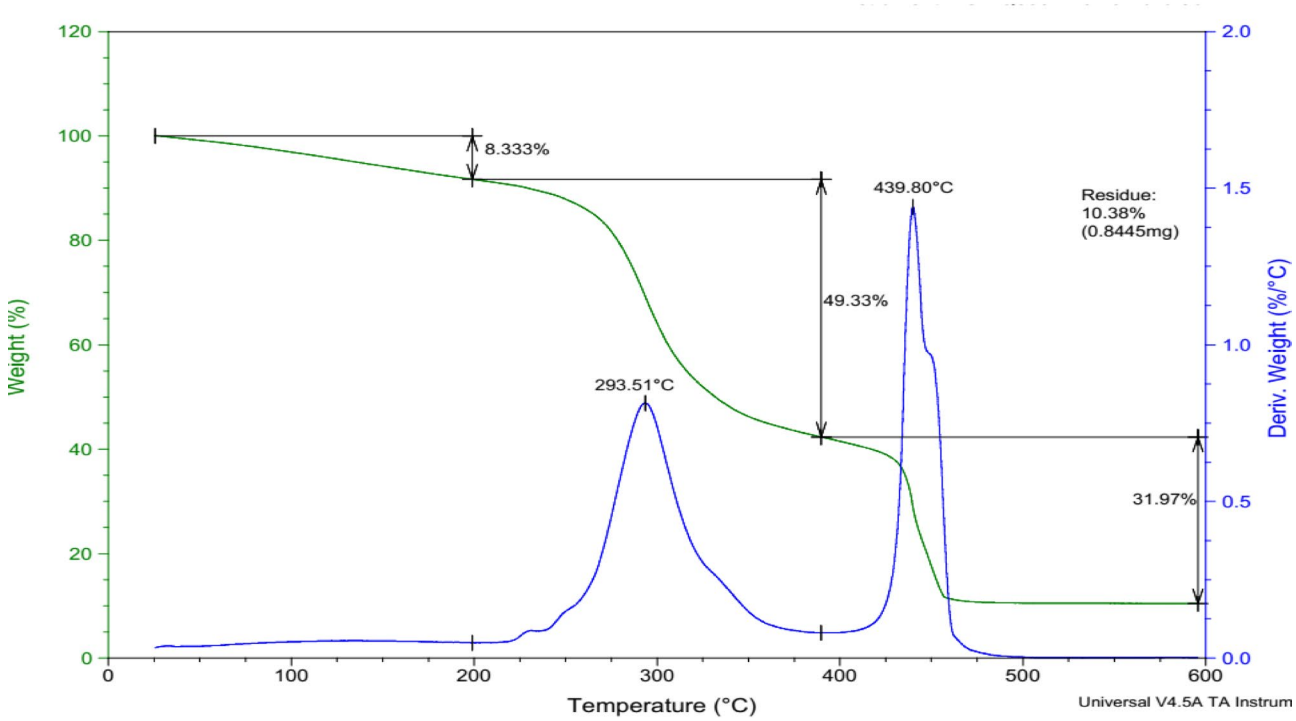


Fig. 2 IR profiles of pulp and seed sampled biomasses



a TGA/DTG of *T.d.* pulp biomass



b TGA/DTG of *T.d* seed biomass

Fig. 3 a TGA/DTG of T.d. pulp biomass. b TGA/DTG of T.d seed biomass.

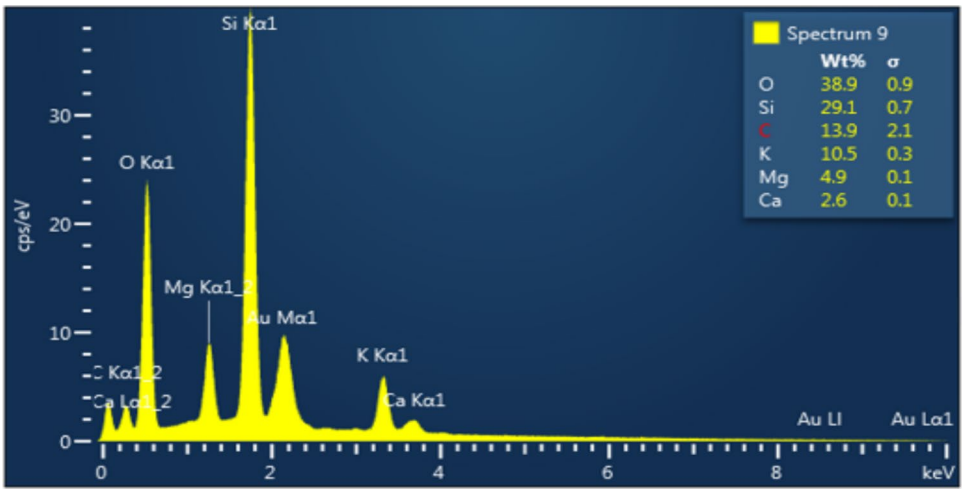


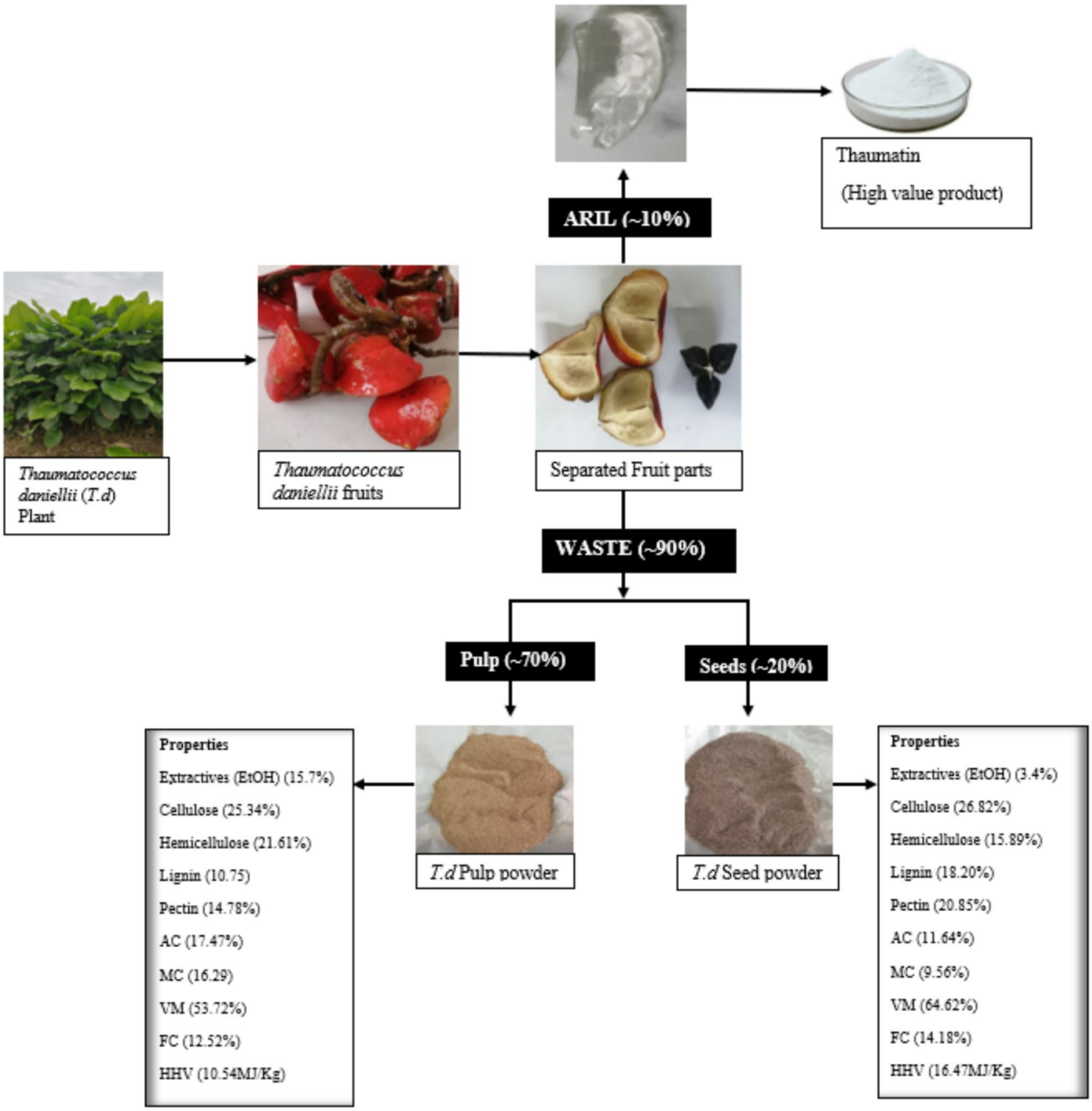
Fig. 4 EDS of seed biomass sample

3.9 Thermal analysis of the biomass

The pyrolysis process, which efficiently converts lignocellulosic biomass into various eco-friendly and value-added products, was evaluated by thermogravimetric (TG)/differential thermogravimetric (DTG) analysis. In addition, predictions of product quality associated with thermal degradation behaviours of sample biomass materials were established. Figure 3a and b show detailed TG/DTG analyses of the pulp and seed sampled biomasses, respectively. The thermogravimetric profiles of the samples show three portions of weight loss due to dehydration, devolatilisation and char formation. The first weight loss attributed to dehydration was observed at temperatures (T) < 200 °C. A closer look at this region shows that the weight loss was divided into two segments. The first weight loss of 8.03% at a temperature range of 25 – 160 °C attributed to water vapour, and the second weight loss of 2.91% at a temperature range of 160 – 201 °C attributed to calorific volatile molecules such as: sugars, lipids and pigments [42, 65]. The second weight-loss portion attributed to devolatilisation was observed within a temperature range of 200 – 400 °C and recorded the highest weight loss for both the pulp (61.77%) and seed (49.33%) samples. The huge weight loss is associated with the pyrolysis of fermentable sugars in the samples, as the aliphatic chains are dissolved while small gaseous molecules are released. The final weight-loss portion attributed to char formation was observed at temperatures T > 400 °C. The weight loss in this portion was also significant (17.48%) for pulp and 31.97% for seed samples and was attributed to the thermal degradation of hydroxyl phenolic and lignin components. The lignin obtained in this study degraded at relatively lower temperatures (400 °C) compared to 500 °C reported in the literature [42]. It suggests that the samples under study are more readily pyrolysed.

3.10 Mineral content of the biomass ash

The EDS results presented in Fig. 4 show high levels of silica (29.1%) in the seed residues, signifying high deposition propensity. The high amount of alkali (K = 10.5%, Ca = 2.6% and Mg = 4.9%) in seed sample biomass could generate ash-related issues in boilers, from molten salts via condensation and vapourisation in addition to a high acid/base ratio [66]. Similar results were obtained by other research groups and indicate that potassium (K) could serve as a catalyst to increase the reaction rate in the movement of free carbon to active sites [67]. Pyrite and quartz were completely absent, indicating that these biomasses as well as their deposits are less corrosive and less destructive than conventional fuels. Furthermore, there is the possibility of a chemical reaction between potassium (K) and silica present in deposits, which may lead to the generation of clingy fluid. Clingy fluids have been reported to be responsible for blocking the air routes of boilers and furnaces [68]. The results obtained from this study are in agreement with those of other analysed biomass samples [42, 56, 69, 70]. In line with the H/C ratios for the *T.d* biomass in this study, the elemental content of the ash of this biomass suggests that the biomass samples could be suitable for the production of biochars which often contain mineral (ash) components such as K, Ca and Mg, which allow biochars to serve as a direct source of mineral nutrients favouring plant growth. These mineral elements are often present in these biochars as carbonates or oxides which give the biochars the ability to be used in the sorption of heavy metals through the formation of precipitates [71]. Additionally, the presence of silicon in biochars has been found to significantly reduce the effect of potentially toxic elements such as Ni in soil, on plants [72].



Scheme 1 Summary of properties of *T.d* biomass samples displaying their potential for biorefinery

4 Conclusion

This study has successfully carried out preliminary characterisation of the *Thaumatococcus daniellii* fruit as a potential biomass source for the biorefinery industry. The results obtained demonstrate that waste from Thaumatin production (pulp and seed) are a potential source of biomass for biorefinery, for augmenting energy and material supply and move towards meeting global demand. The huge quantities of biowaste generated from the process as well as their properties, make these biomass samples potential feedstock for sustainable conversion to bioenergy and bioproducts in biorefineries and simultaneously clean the environment and eliminate the possible pollution problem as presented in Scheme 1. Therefore, the goal of valorising these waste biomass samples and eliminating them from the environment has been accomplished, while adding value to the *Thaumatococcus daniellii* fruit.

Author contribution The authors confirm contribution to the manuscript as follows: study conception, laboratory experiments, data collection, interpretation of results, draft manuscript preparation: [Lena Yoh Ekaney Elango]; chemical analysis, draft manuscript preparation, review and editing: [Henrietta Wakuna Langmi]; study design, data collection and draft manuscript preparation: [Victorine Beckley Namondo]; results interpretation, analysis and preparation of draft manuscript: [Ekane Peter Etape]; study design, manuscript preparation, review and editing: [Jane-Francis Akoachere]; Study conception, design, manuscript preparation, review and supervision: [Josepha Foba-Tendo]. All authors read and approved the final version of the manuscript.

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