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Physico-chemical Characterization and Qualitative Analysis of Phytochemicals Present in Food Additives Derived from *Sesamum indicum*, *Brassica nigra* Plant Wastes and *Cocos nucifera* Husk

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ABSTRACT
Agricultural food sources and their plants have a lot of utilization for the production of food as well as other related products. Indigenous ways of preparing food products are adopted by the industries and those have high commercial importance. In Assam, a northeastern state of India, native people prepare a food additive from *Sesamum indicum*, *Brassica nigra* plant waste, and *Cocos nucifera* husk which is popularly known as 'Khar' or 'Khardwi'. The water extract of the ashes of those plant wastes is used as an alkaline food additive. In this work, the physicochemical characterization of the ashes and the extract of the ashes were done using various standard analytical methods. The pH of these extracts is in the range of 10-12 and contains high amounts of K, Na, Ca, and Mg. The presence of phytochemicals was also found in the preliminary investigation. Other essential minerals like Fe, Mn, Cu, and Zn enhance the quality as a food additive.

Keywords: Black benni seed, Mustard, Coconut, Khar, Ash water extract

INTRODUCTION

Food additives have a great role in food science and people find different food additives according to their food culture, geographical conditions, availability of resources and sometimes depending on the situation created by the body after eating of food. In Assam, a Northeastern state of India, people have the habit of eating a lot of fish and meat. These foods cause acidity for which, people here have developed an alkaline food additive which is applied to fish, meat, vegetables, etc. during preparation. In the Assamese language, it is called 'Khar'and the Bodo tribe of Assam is called 'Khardwi' which is obtained mainly from plant waste (Deka 2007, Mudiar et al. 2014). These alkaline food additives have been used by the Assamese people in their food since ancient times. Apart from its use as an additive, it has also been used to treat various illnesses (Mudiar et. al. 2014, Kalita and Bikash 2004, Debabandya 2010, Hussain 2010). Conventionally, it is used to control digestive disorders of the stomach and to avoid bacterial attacks on freshly cut injuries to heal fast. In village areas of Assam, people use 'Khar' as an alternative to soaps and detergents for washing clothes and cleansing hair from long back. Farmers usually use it while working in water-logged field areas to get rid of leeches as bio-insecticide, as it can avoid the attack of leeches. The 'Khar' is a unique dish of Assam, which is served in Assamese meals and it is believed by the Assamese people that it purifies the stomach.

Traditionally it is prepared from the different parts of the banana plant by filtering the ash extract of various parts of the dried plant like pseudo-stem, rhizome, and peel. Since the ash of banana waste has enormous uses and the extract is beneficial as a food additive, people of Assam have used other plant-based ashes for the same purpose (Kalita and Bikash 2004, Debabandya 2010, Hussain 2010, Kadhirvel 2010, Arawande and Komolafe 2010, Adinarayana and Babu 2011, Preeti 2011, Mudiaret al. 2014).

In Assam, India, the different tribal and non-tribal communities extensively cultivate Mati Dal (Black gram lentils, *Vigna mungo*), Till (black benni seed, *Sesamum radiatum*), and Mustard plant (*Brassica*

nigra). After separating the seed; the plant materials are generally thrown away as waste materials that have no commercial value. The waste plants are dried under sunlight and after removing the soil particles they are burnt thoroughly in the open air and the ashes thus obtained are used as an insect killer in crop fields. It is important to mention here that the water extract of ash obtained from all these plants is highly alkaline which is used as Khar or Khardwi. Similarly, a Coconut (*Cocos nucifera*) shell is also burnt and used to prepare this Khar.

The objective of the present work is to study the physicochemical characteristics of the Khar liquid and the plant ash by using different laboratory analyses and instrumental methods. The surfaces of the ash particles are analyzed by using SEM-EDX and functional group analysis by using FTIR. This extract may have some other good properties like antioxidant behaviors, because of the presence of phytochemicals present in these compounds. Therefore, the qualitative analysis of the phytochemicals was done using analytical methods.

MATERIALS AND METHODS

The ashes of black benni seed (Till) and Mustard plant were collected from the local market where the native women used to sell them. The coconut husk was collected, ash was prepared in the laboratory and the extract was prepared using distilled water and following the same procedure by the native people (Fig. 1). The ash was kept with distilled water overnight and filtered the next day to get the extract. Other chemicals required for the analytical purpose were taken from Thermo Fischer Scientific of AR grade.

The native people don't use the exact measured amount of ash to make the extract. They use the dried half of the coconut shell where a small slit is made at the bottom. Small pieces of dried bamboo sticks were inserted through the slit. The ash used to be taken from the coconut shell to below 3-4 cm from the top. Water is added to the ash making the volume to the top of the coconut shell. The water gets enough time to pass through the fine particles of the ash and

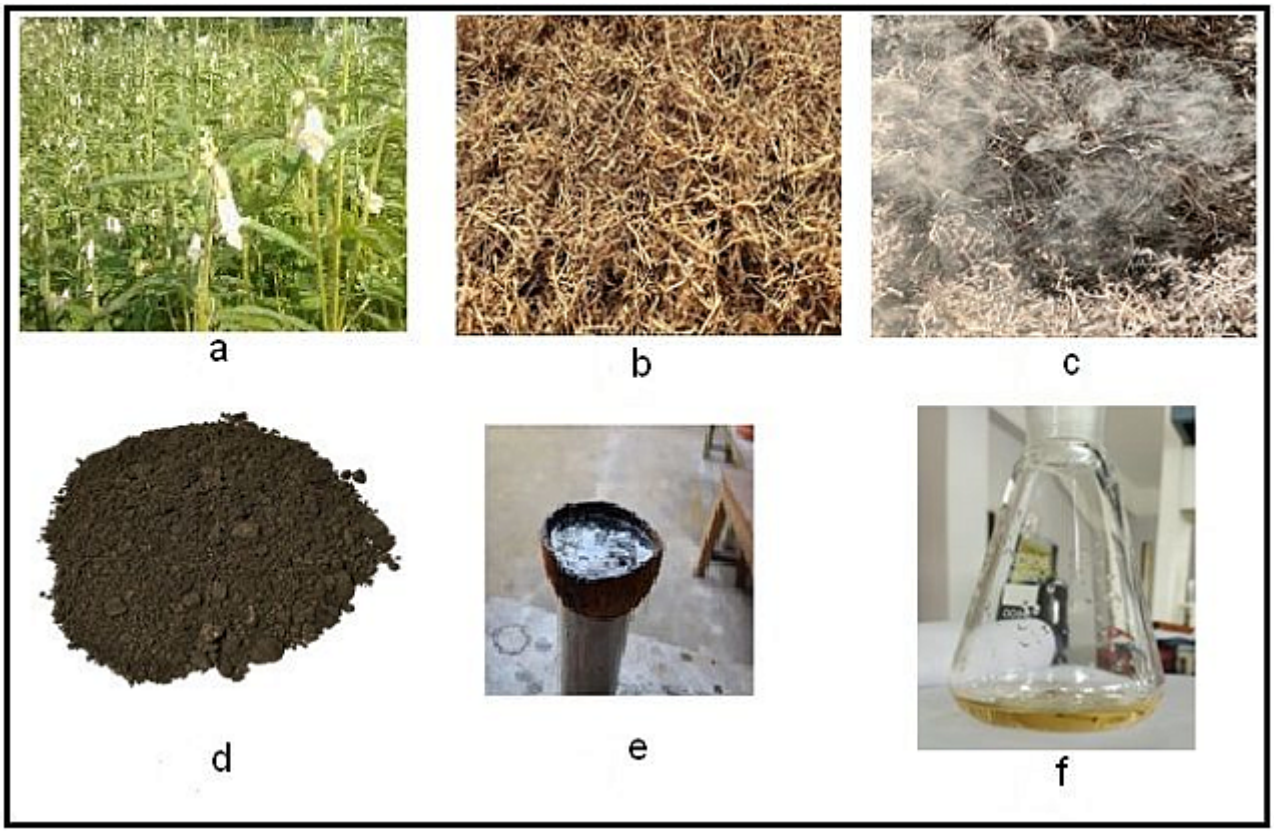

Figure 1. a) Fresh plants of *Sesamum indicum*, b) Seeds and plants washed thoroughly and dried, c) Dried plants are burnt to ash, d) Dried Ash powder, e) Water extract of ash by villagers using coconut shell as a funnel, f) Water extract of ash in lab using Whatman filter paper

it falls very slowly into the glass or other pot which is placed at the bottom of the coconut as seen in Figure 1(e). A similar process was tried in the laboratory preparation of the ash extract; however, after trying different amounts of ash 1g in 100mL water was taken as the best weight of the ash and the volume of water which gives the maximum pH. Labtronic digital pH meter Model No. LT-11 was used for the determination of pH after calibrating the instrument at pH 9.20, 7.00, and 4.00. The carbonate and bicarbonate alkalinity was determined by the double titration method (https://archive.epa.gov/water/archive/web/html/vms510.html) and the hardness was measured by applying the complexometric titration method (https://www.nemi.gov/methods/method_summary/4684/). Atomic Absorption Spectrometer (AAS) Thermo Scientific model no.: ICE3000 Series was used for the quantitative analysis of different metals. FTIR of asheswas studied using the Perkin Elmer SPECTRUM 100 model. The surface morphology of the ash sample was studied by using SEM (scanning electron microscope) JEOL, JAPAN, model no. JSM 6390LV. The percentage of different elements was determined by the EDX (Energy Dispersive X-Ray Analysis) technique.

RESULTS AND DISCUSSION

The water extract of the ashes is characterized by using different analytical techniques which are presented here. The results of different physicochemical parameters done by titration and instrumental methods according to standard procedure are presented in Table 1. The metal element content of the extract of ashes is presented in Table 2. Figure 2 shows the surface morphology of the ash from SEM micrographs and the presence of different elements in the solid ash samples can be observed from the EDX data presented in Table 3. The presence of different functional groups can be observed from the FTIR spectrum in Figure 3.

Physio-chemical parameters

The water extract of the ashes of *Sesamum indicum*, *Brassica nigra* plant waste, and *Cocos nucifera* husk are highly alkaline which can be correlated with high carbonate and bicarbonate content. This is why the native people use those as basic food additives learning from their experience (Deka 2007). However, the method of preparation of the extract is strictly based on the experience of people forwarded to the new generation by the older. Therefore, sometimes the same sample may give a different pH within the range of 10 to 11. To avoid this situation, in this work a range of extracts was prepared using different weights of the ash sample and water and tested for pH. For example, 0.1, 0.3, 0.5, and 1.0 g per 10 to 100 mL of water and the pH of each solution was measured and a good correlation was found between the weight of ash and the water added. So, a common weight of the ash and volume of water i.e. 1 g of ash per 100 mL of water was selected for all the samples and accordingly, the pH values are presented in the table.

This amazing liquid is also an alternative source of common salt used by the tribes here from their traditional knowledge. Surprisingly, the liquid extract contains a lot of potassium and sodium which is generally taken in the diet by using common salt. The hardness of the extract is a little high may be due to the presence of fine clay particles with ash

Table 1. Physicochemical parameters

Parameter	*Sesamum indicum* (Til)	*Brassica nigra* (Mustard)	*Cocos nucifera* (Coconut)
pH (1g ash/100ml DW)*	11.34	10.90	10.77
Alkalinity (Bicarbonate)	1336 ppm	720 ppm	880 ppm
Alkalinity (Carbonate)	1136 ppm	980 ppm	1800 ppm
Total hardness	224 ppm	780 ppm	124 ppm
TDS	438.8ppm	672.3ppm	432.9ppm
Chloride	4.49ppm	17.25ppm	10.75ppm

*DW=Distilled Water

Table 2. Presence of metal elements

Element (in ppm)	*Sesamum indicum* (Til)	*Brassica nigra* (Mustard)	*Cocos nucifera* (Coconut)
K	27142.8600	28977.9900	360157.7000
Mg	336.6550	597.7500	0.0689
Na	107.1600	251.4500	4694.5400
Ca	9.6518	7.0181	6.9615
Cu	0.3358	0.6190	0.2215
Mn	0.1045	0.2655	0.0771
Fe	0.0872	0.0827	0.0592
As	BDL	BDL	BDL
Zn	BDL	BDL	BDL
Pb	BDL	BDL	0.0313
Ni	BDL	BDL	BDL

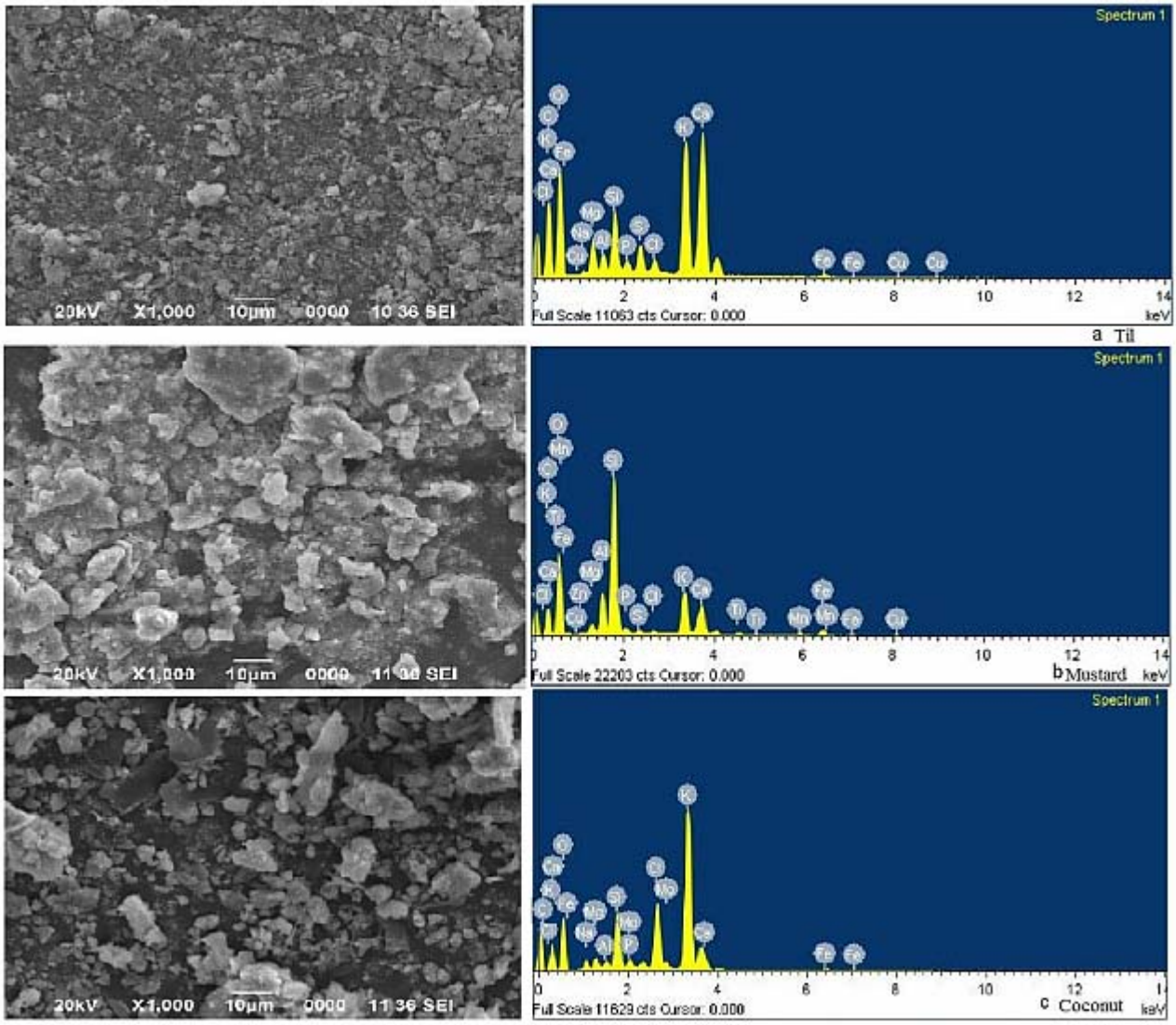

Figure 2. SEM-EDX spectrum of the ash samples showing the concentration of different elements

due to the lack of proper separation of soil materials from the dried plants which is different in the case of Coconut shell ash extract.

However, this may not affect the food prepared since only 5-10 mL of the extract is used for food-making purposes; this value will further decrease in the food prepared. However, these concentrations may vary according to the weight taken for the extraction process. Therefore, it is necessary to determine the concentrations of different metals present in the samples of the extracts using an Atomic Absorption Spectrophotometer (AAS).

Elemental analysis

The metals present in the samples of extracts are done by using AAS as shown in Table 2. The highest amount of potassium is followed by other metals K > Mg> Na> Ca > Cu > Mn > Fe and Zn, Pb, As, and Ni metals were not found. A very high amount of K and Na are found in the Coconut shell ash extract. The necessity of these metals for human health is well known in the new world. Surprisingly, the common people have learned the benefit of this liquid extract from their ancestors as this method of preparation of KHAR was more than two hundred years ago. In the coconut sample trace amount of Pb was found, however in the SEM-EDX analysis, this metal was not observed in the ash. In previous work on banana ash extract, a small amount of Pb was found (Deka 2007). So, it may be correlated with the water, which may have a trace quantity of Pb.

The AAS analysis reveals that the metal nutrients present in the extract are very beneficial for human health and the alkalinity of the samples shows its efficacy more in terms of gastric related disorder; therefore, people in Assam traditionally take one spoonful of the extract with a glass of water to overcome the problem. This may be an example of herbal medicine with no side effects.

SEM-EDX analysis

The SEM-EDX analysis offers the surface analysis and the elemental composition shown in Figure 2 and Table 2. The SEM micrograph shows almost uniform fine particles on the surfaces of the ashes because after burning, the plant-dried materials are sieved through fine bamboo-made sieves, and only the fine parts are collected. These ashes may have potential application in the removal of heavy metals from wastewater as it was done for banana ash (Das et al. 2007, Bordoloi et al. 2011). From the EDX analysis, it is clear that K, Mg, Ca, Fe, and Si are the main metals present in the ashes followed by some other elements. The AAS analysis (Table 2) of the extract also confirmed the existence of K at the highest concentration which is followed by Mg and Na. Low levels of Cu, Mn, Fe and Cu were also evident from the AAS study which is in agreement with the EDX report. However, there are some mismatched values of concentration of the elements as obtained by SEM-EDX and AAS techniques because SEM-EDX was done with the ash samples and AAS was used for the analysis of the water extract of the ash. The ash samples may have the chance of having clay particles which is reflected in the amount of Si present, because of the procedure of preparation.

EDX is a qualitative and semi-quantitative technique used for the detection of elemental distributions. It is important to note that EDX analysis is generally done at different sites or locations of the sample where the elements may not be uniformly distributed due to which the concentration of elements detected at a particular site may not be the same at other sites of the sample. This might be the cause for the difference in results with that of the AAS reports since AAS is a quantitative tool for liquid samples.Thus, the characterization confirmed the presence of high concentrations of K, Mg, Na, and Ca for which these ash extracts can be employed in different experiments as a base (Boruah et al. 2015).

FTIR assessment of ash

The FT-IR spectrums of the ashes are shown in Figure 3 and it is observed that the broad peak appears around 3400 cm^{-1} which indicates the presence of the -OH group due to the water molecules adsorbed on the surface of the ashes. The small peak at 2400 cm^{-1} is due to M-O stretching vibration which indicates the presence of potassium in the ash samples and this is in agreement with the IR peak (2178 cm^{-1}) of *Musa acuminata* peel catalyst (Pathak et al. (2018). The presence of peaks at 1640 cm^{-1}, and 1398 cm^{-1} indicate C-O stretching vibrations due to the presence of metal carbonates in the catalyst

Table 3. Percentage of different elements present in the ash samples

Element	Till		Mustard		Coconut	
	Weight	Atomic	Weight	Atomic	Weight	Atomic
C	44.68	56.97	23.96	34.79	22.41	29.35
O	36.53	34.96	43.94	47.88	35.42	50.65
Mg	1.2	0.76	0.93	0.67	1.55	0.46
Al	0.58	0.33	3.46	2.24	0.76	0.64
Si	1.99	1.08	13.96	8.66	6.76	2.52
P	0.43	0.21	0.66	0.37	1.03	0.76
Cl	0.43	0.19	0.3	0.15	9.33	3.04
K	5.89	2.31	5.35	2.39	15.9	9.31
Ca	6.93	2.65	3.93	1.71	2.05	1.17
Fe	0.38	0.1	1.67	0.52	0.7	0.11
Ti	0.15	0.05	0.28	0.1		
S	0.81	0.39	0.29	0.16		
Cu			0.43	0.12		
Zn			0.37	0.1		
Mn			0.45	0.14		
Mo					2.3	0.21
Na					1.79	1.78
Total	**100**		**100**		**100**	

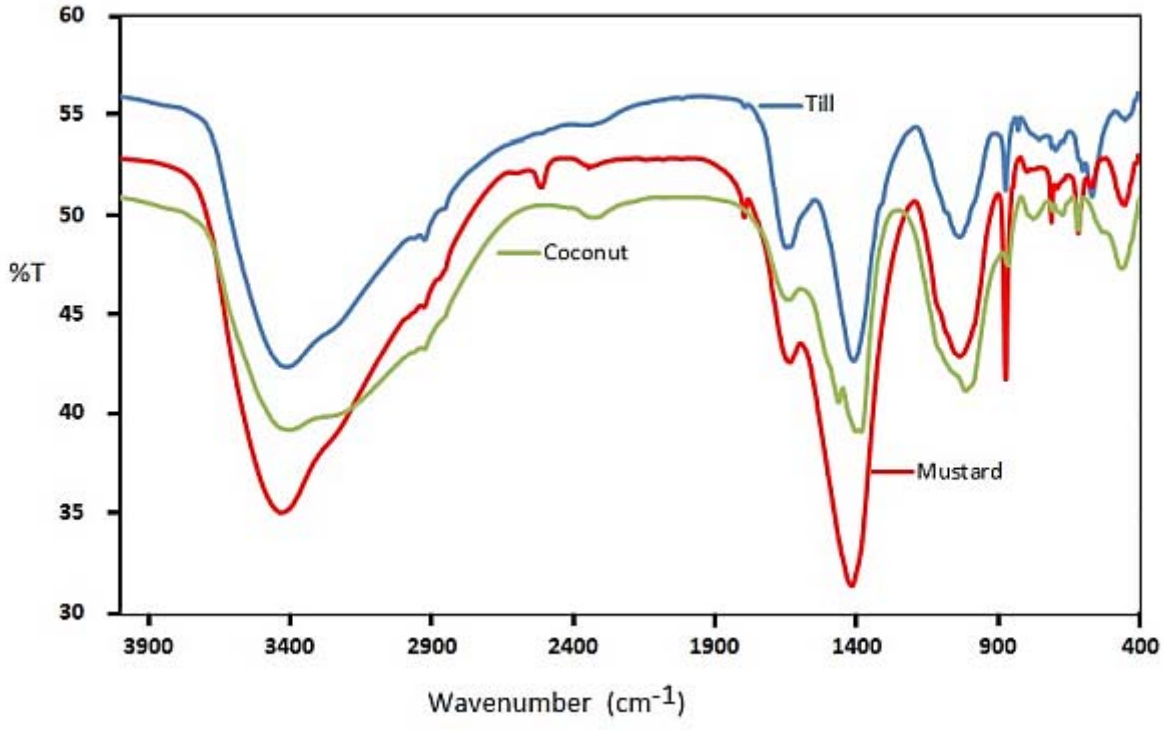

Figure 3. The FT-IR spectrum of ashes

which is in agreement with the FT-IR analysis of catalysts reported by Gohain et al. (2017) and Pathak et al. (2018). The peaks at 1012 cm^{-1} and 774 cm^{-1} may be due to the Si-O-Si bond. The peak at 615 cm^{-1} to the K-O and Ca-O stretching vibrations may be due to the presence of K_2O and CaO. Similarly, Pathak et al. (2018) also reported an FT-IR peak at 687 cm^{-1} for K-O and Ca-O stretching vibrations in the *Musa acuminata* peel catalyst. In this study, the FT-IR analysis is in satisfactory agreement with the EDX data which confirmed the presence of oxides and carbonates of metals in the samples.

Phytochemicals

All plant extracts have phytochemicals that may be useful for different purposes (Shaikh and Patil 2020, Kalita et al. 2015, Kumar et al. 2023). Since the production of these plant extracts has been done after burning the dried plant materials, therefore, different phytochemicals that were present in the plants may not be present in the ashes. So, a qualitative analysis of the phytochemicals is done according to the standard procedure (Shaikh and Patil 2020, Kalita et al. 2015). The results of the tests for the phytochemicals are presented in Table 4. From the table, it can be observed that the alkaloids, carbohydrates, and saponins are present in all the samples where as phlobatanies, terpenoids, and steroid is absent. In coconut ash extract, the highest number of phytochemicals are present, followed by mustard and til. However, depending on the sample source it may vary to some extent. This may be emphasized here that even after burning the plant materials some phytochemicals are still present in

Table 4. Different phytochemicals present in the water extract of the plant ashes

Phytochemicals	**Til**	**Mustard**	**Coconut**	Activities (Shaikh and Patil 2020)
Tannins	-	+	+	Cardioprotective, anti-inflammatory, immunemodulatory, anti-carcinogenic and anti-mutagenic
Alkaloids	+	+	+	Antiviral effects
Cardiac Glycosides	-	-	+	Treatment of cardiac failure, anti-cancer activities
Steroids	-	-	-	Antihelminthic, antibacterial, anti-tumor activities and growth hormone regulation
Phlobatannins	-	-	-	Anti-inflammatory and analgesic and antioxidant
Amino Acids	-	-	+	Muscle development and strength
Carbohydrates	+	+	+	The body's main fuel source
Fixed oils and fats	-	-	-	Veritable source of energy, boost the immune system
Glycosides	-	+	-	Allelopathic, antiseptic effects
Phenolic Compounds	+	+	+	Potential antioxidants and avert the damage of cells resulted from free-radical oxidation reactions
Phytosterols	-	-	-	Lowering cholesterol
Saponins	+	+	+	Decrease blood lipids, lower cancer risks, and lower blood glucose response
Gums and mucilages	-	+	+	Acting as biopolymeric encapsulating agent
Terpenoids	-	-	-	Antitumor, anti-inflammatory, antibacterial, antiviral, antimalarial effects
Flavonoids	-	+	+	Anticancer, antioxidant, anti-inflammatory and antiviral properties

the samples of the extracts. This may add another important aspect of these food additives for the said purpose.

CONCLUSIONS

The *Sesamum indicum* (Til), *Brassica nigra* (Mustard) plant, and *Cocos nucifera* (Coconut) shell husk ashes are used for the preparation of an extract which is used mainly as a food additive for the preparation of a dish called Khar in Assamese language. The ashes or the extract have other uses such as controlling gastric disorders, manure, paste controller, and many other purposes by the natives here. The extracts have a high pH in the range of 10-12 which is correlated with the high amount of K, Na, Ca, and Mg or their carbonates and hydroxides. AAS data shows that all essential metals are present in the extracts which are a regular requirement of the diet. A homogeneous surface is observed in all the ashes through SEM analysis and agglomeration of particles in different ratios has been seen which may be due to the moisture content. The ash may be used as an adsorbent for heavy metals, dyes, or organic pollutants as well as for other applications in chemical science. C-O, M-C, and M-O vibrational frequencies are observed in the FTIR analysis. K, Na, Ca, and Mg metals are present in higher amounts whereas the presence of essential minerals like Fe, Mn, Cu, and Zn enhances the quality as a food additive. The ash extract may have some antioxidant activity because of phytochemicals, so this may have some future scope of study.

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