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Research Article

## Cassava peel wax: its extraction and characterisation

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**Abstract:** Cassava peel has become conspicuous environmental waste which affects the natural integrity of the environment. Several researchers have extracted wax from agro-wastes such as sugarcane bagasse, banana peel, apple pomace etc. The present study extracted wax from cassava peel using soxhlet extraction method with a mixture of benzene and methanol as the extracting solvents. The percentage yield of crude wax from cassava peel was 8.51%. The wax extracted was characterised using fourier transformed infrared spectroscopy (FT-IR), ultraviolet-visible spectroscopy (UV-Vis) and scanning electron microscopy (SEM). The physicochemical properties were studied; acid value, saponification value, ester value, melting point and iodine value. The FT-IR prominent peaks were obtained at 3224(-OH), 2928 (-CH<sub>2</sub>), 1376.96 (-CH<sub>3</sub>), 1708 (-CHO), 1035 and 885, indicating presence of alcohols, alkanes, aldehydes, and lignin compounds, respectively. The UV-Vis analysis showed fatty acids, conjugated dienes, and hydroperoxides at 232 nm and conjugated trienes at about 255 nm. The results of the physicochemical properties were evaluated; acid value, saponification value, ester value, melting point and iodine value as 23.4 mg/KOH/g, 103.42 mg/KOH/g, 80.02 mg/KOH/g, 63.5°C and 90.3 g/100g, respectively. This study revealed that wax obtained from cassava peel is a potent product for diverse industrial applications.

**Keywords:** Cassava peel, isolation, characterisation, agro-waste, wax

### INTRODUCTION

Cassava peels are agro-waste resources discarded after peeling the tuber. It can represent 5 to 15 % of the root.<sup>1,2</sup> They are obtained after the tubers have been water-cleansed and peeled mechanically<sup>1</sup>. They may contain high amounts of cyanogenic glycosides and have higher protein content than the tuber parts<sup>3</sup>. Cassava peel wax was extracted using soxhlet extraction method with a mixture of benzene and methanol. The wax portion finds its applications in cosmetics, paper coating, textiles, fruit and vegetable coating, leather sizing, lubricants, adhesives, polishes and pharmaceutical industries.

Hence, it can be considered as an alternative to costly carnauba wax, candelilla wax and chitosan for edible coating applications. Ash gourd peel was found as a good source of wax which was used as edible coat for strawberries to enhance their shelf life<sup>4</sup>. The present study was aimed at adding value to cassava by using their peels as raw material to extract wax. This also proffers solution to the problem of indiscriminate disposal of peels of cassava, which creates nuisance in the environment. The utilization of cassava peel (agricultural wastes) into wax is considered a potent product for various industrial applications.

## MATERIALS AND METHODS

**Plant Material:** Cassava peels (agro-waste) were collected from a local cassava processing settlement. The sample was stored in sacks adequately. The sample was washed to remove dirt and dried to a constant weight in a hot air oven at 80°C and was triturated in a mill into fine powder and stored in plastic containers.

**Wax Extraction:** Wax was extracted from cassava peels as reported by Anuj *et al.*<sup>5</sup> The cassava peel powder was extracted using soxhlet extraction method with benzene and ethanol mixture of 2:1 v/v for 8 h to remove wax. The solvent was later evaporated using rotary evaporator (Buchi, Switzerland) and the residue primarily contained wax mixtures with some impurities. The residue was dissolved in isopropanol and was refluxed with charcoal for 1-2 h to remove any undesirable colour or pigments present. The extract was then filtered to remove the charcoal and evaporated at 60°C to obtain crude wax.

**Physicochemical Analysis:** The physicochemical analysis was carried out according to the method by Mangesh and Lele<sup>6</sup>.

**Acid value:** 5.0 g of wax was weighed into a dry conical flask, 25 ml of absolute ethanol and 2 drops of phenolphthalein indicator were added to the mixture. The mixture was heated with continuous shaking in water bath for 10 min and cooled. The cooled mixture was titrated against 0.1 N KOH solution until pink colour appeared. The acid value was calculated

**Saponification value:** 2 g of the wax sample was weighed into a 250 ml conical flask and 25 ml of alcoholic potassium iodide was dissolved in it. A reflux condenser was attached and the flask content was heated on a boiling hot water for 1 h with occasional shaking, 3 drops of phenolphthalein indicator was added and then titrated with 0.5 N HCl. This was repeated for blank sample.

**Iodine Value:** 2 g of the wax was weighed, 10 ml of chloroform and 30 ml of Hannus solution was added into a 250 ml conical flask, the flask was sealed with parafilm and the solution was agitated for 30 min. A mixture of 10 ml of 15% potassium iodide and 10 ml of distilled water was added. The iodine solution was titrated against 0.1 N sodium thiosulphate solution using starch as an indicator. This was repeated for blank.

**Melting Point:** The melting point of cassava peel wax was done according to the method adopted by Bekele *et al.*<sup>7</sup>. The wax was melted by warming it in a hot water bath and the melting point was determined by the capillary tube method. Melted wax was introduced in a 10 cm long and 2 mm internal diameter thin-wall

hollow capillary tube, until reaching a height of about 1 cm. After that, the capillary tube, containing the wax, was introduced into a bath of water that was slowly warmed at 1–2°C/min; the temperature was studied with a thermometer (with an accuracy of 0.1°C) whose bulb had to be as close as possible to the beeswax column introduced in the capillary tube.

#### Analytical Methodologies:

**Fourier Transform Infrared Analysis:** Wax sample was placed on the diamond crystal and the ATR holder was screwed onto the sample to have a good contact with the crystal. The PerkinElmer spectrum 400 FT-IR spectrometer was set to scan 60 times at a resolution of 2.0 cm<sup>-1</sup> over the wavelength range of 4000 cm<sup>-1</sup> to 650 cm<sup>-1</sup> against the transmittance. After every scan, the baseline was corrected using the background spectrum previously obtained from the blank.

**UV-Vis analysis:** A known quantity of wax (0.1% w/v) was dissolved in hexane for spectroscopic measurement. The solvent was scanned across wavelength of 200 to 600 nm by using Helios  $\alpha$  spectrophotometer (Thermo Electron Corporation, USA).

**Scanning Electron Microscopy (SEM):** In this study AURIGA Field Emission High Resolution Scanning Electron Microscope was used to analyse the surface morphology of the raw and dewaxed cassava peel. The samples were prepared by coating with carbon to make SEM work conductive; Emitech K950X Carbon evaporator was used for the coating process.

## RESULTS AND DISCUSSION

**Cassava Peel Wax Yield:** The yield of wax was found to be 8.51% of dried cassava peels. This yield was higher compared to that reported by Mangesh and Lele<sup>6</sup>, which was found to be 0.95% (w/w). This could be as a result of the selective extractability of the main components in wax- a mixture with different polarities (mass ratio of benzene and methanol, 2:1) used as this mixture could efficiently extract both polar and nonpolar compounds and give a high wax yield from cassava peel. **Table 1** presents the physicochemical properties of crude wax from cassava peel.

**Table (1):** Physicochemical Properties of Crude Wax from Cassava Peel

Physicochemical Parameters	Cassava Peel Wax
Odour	Agreeable odour
Nature	Viscous
Melting point (°C)	63.5
Acid value (mg/KOH/g)	23.4
Saponification value (mg/KOH/g)	103.85
Iodine value (g/100g)	90.05
Ester value (mg/KOH/g)	80.45

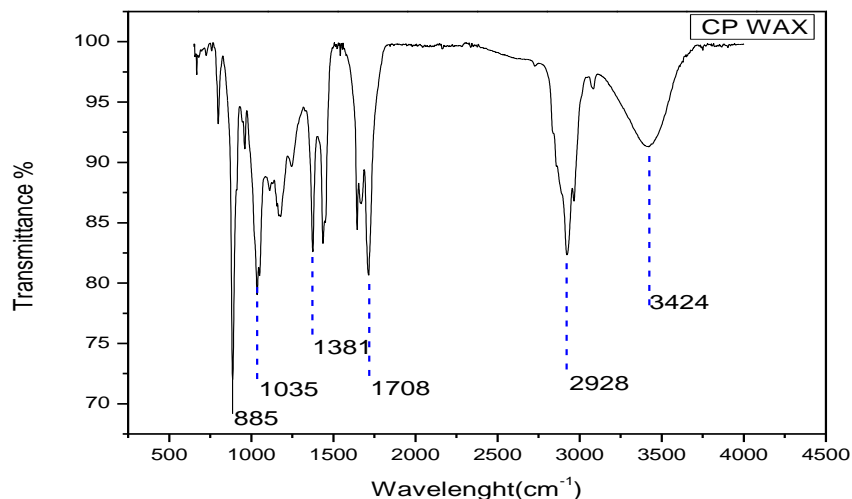
**Physicochemical Analysis:** **Table 1** shows the results of the physicochemical parameters of the wax extracted from cassava peel. In this present study, wax from cassava peel, being crude in nature showed melting point comparable to those from Mangesh and Lele<sup>6</sup> and Bekele *et al.*<sup>7</sup> which were 62.12 and 62.39 ± 0.12, respectively, however, it was found to be higher than that reported by Athukorala<sup>8</sup> which was 55-56.

The acid value, saponification value and ester value was lower compared to that reported by Mangesh and Lele<sup>6</sup>, which was 129.15, 374.58 and 245.08, respectively but was comparable with that reported by Athukorala<sup>8</sup> with  $22.33 \pm 0.39$ ,  $98.04 \pm 0.86$  and  $75.58 \pm 0.84$ , respectively.

**FT-IR Analysis:** Figure 1 and Table 2 presents the FT-IR spectra of wax from cassava peel. FT-IR analysis of wax sample revealed the presence of several organic functional groups present in crude wax indicating their respective compounds. A broad and blunt IR absorption band was observed in the region of  $3424 \text{ cm}^{-1}$ , this reveals that the wax possesses hydroxyl group O–H stretching. The strong absorption bands at  $2928 \text{ cm}^{-1}$  indicates the presence of –CH stretch and bend while  $1381 \text{ cm}^{-1}$  reveals the presence of  $\text{CH}_3$  group, which are characteristics of aliphatic fraction of the wax. Similarly, bands at  $1708 \text{ cm}^{-1}$  and  $1035 \text{ cm}^{-1}$  showed the presence of –C=O stretch but represented two different functional groups; aldehydic and carboxylic acid and it is also assigned to carbonyl stretching in fatty acids. The FT-IR spectra of cassava peel wax exhibited similar absorption patterns with those reported by Athukorala<sup>8</sup>, Knuutinen and Norrman<sup>9</sup> and Bekele *et al.*<sup>7</sup>.

**Table (2):** FT-IR Spectra of Crude Wax from Cassava Peel

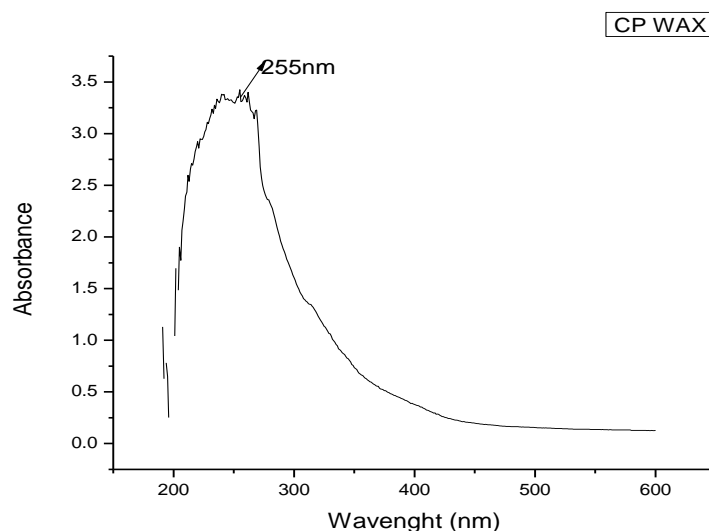
Band position	Assignment
3,443	O–H stretching vibration (associated to water or alcohols)
2,928	Asymmetric CH <sub>2</sub> stretching (associated to aliphatic fraction)
1,708	Carbonyl stretching in fatty acids
1,381	CH <sub>2</sub> bending vibrations (associated to lignin or wax)
1,035	C–H deformation in guaiacyl unit (associated to lignin)
855	Aromatic C–H out-of-plane vibrations (associated to lignin)



**Figure (1):** FT-IR Spectra of Crude Wax from Cassava Peel

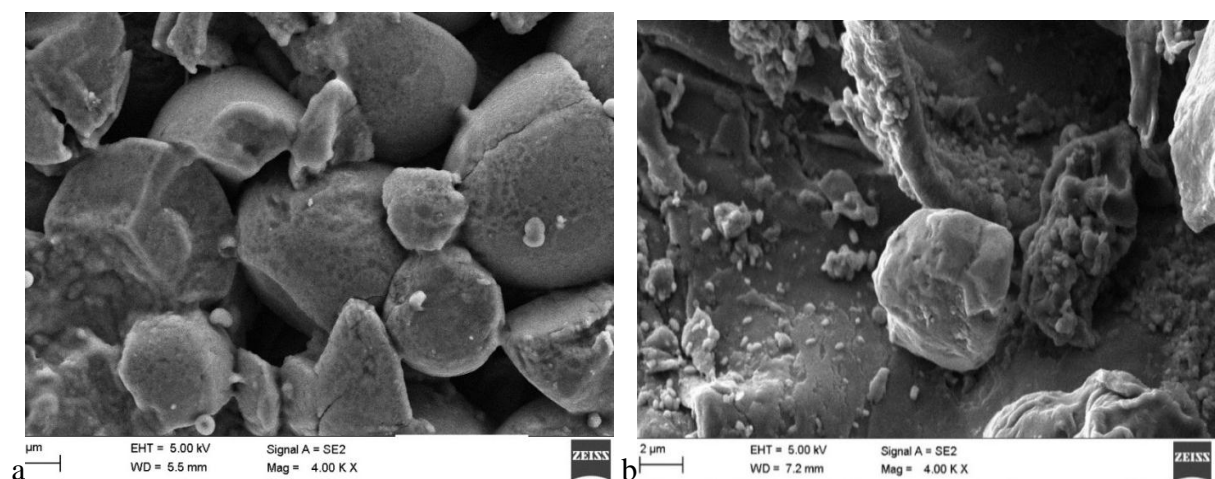
**UV-Vis Analysis:** Figure 2 presents the UV-Vis Spectra of cassava peel. Fatty acids, conjugated dienes, and hydroperoxides formed as a result of lipid oxidation absorbed UV light at about 232 nm and conjugated trienes at about 255 nm. In the UV range of 100–400 nm, cassava peel wax showed sharp peaks near 255 nm indicating the presence of conjugated dienes and trienes as presented in Figure 2. Also, in 400–600 nm range no peak was found indicating the absence of carotenoids. This result was similar to that reported by Mangesh

and Lele<sup>6</sup>, which showed sharp peaks near 230 and 270 nm indicating the presence of conjugated dienes and trienes. The result of the cassava peel was in agreement with that reported by Athukorala *et al.*<sup>8</sup>.



**Figure (2):** UV-Vis Spectra of Crude Wax from Cassava Peel.

**Scanning Electron Microscopy (SEM):** The surface morphology of native and dewaxed cassava peel was measured and the result was presented in **Figure 3a and 3b** above. Figure 3a shows the surface morphology of native cassava peel, which appears to have a smooth and compacted surface due to the presence of some non-fibrous components in the fibre surface such as lignin, hemicellulose, wax, pectin, oil etc., Figure 3b shows the surface morphology of dewaxed cassava peel, which appears with a rough, cracked and individualised surface indicating the removal of wax. This result was similar to that reported by Gaoxiang *et al.*<sup>10</sup>



**Figure 3:** (a) SEM Micrograph of Raw Cassava Peel (b) SEM Micrograph of Dewaxed Cassava Peel

## CONCLUSION

The yield of crude wax from sugarcane peel was 8.51% (w/w). This study showed the presence of various classes of compounds such as; alkane, ester, alcohol, fatty acids among others, present in cassava peel wax. In the UV range of 100–400 nm, cassava peel wax showed a sharp peak near 255 nm indicating the presence of conjugated dienes and trienes. The SEM micrograph of the dewaxed sample revealed the removal of wax from the fibre. The present of fatty acids played an important part in human nutrition which was found in the wax. The wax extracted had reasonable physicochemical characteristics comparable to those studied in literatures. Thus, cassava peel wax has shown numerous compounds of biological and industrial importance.

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