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Physico-chemical, Functional, Thermal, Dielectric and Surface Characteristics of Freeze-dried Watermelon Rind Powder

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ABSTRACT

Watermelon rind is generally considered agricultural waste and has wide application due to the presence of polyphenolic compounds and citrulline which have many health benefits. This investigation was aimed at the valorisation of watermelon rind by converting it into freeze-dried powder. The powder was examined for its physico-chemical, dielectric, structural, functional, thermal and flow characteristics. Watermelon rind powder had 10.22% moisture, 12.93% ash, 11.16% protein, 1.30% fat and 0.40% water activity, with a slight green tint in colour. It had good solubility, flowability and foaming. Semi-crystalline profile was demonstrated by X-ray diffraction. Scanning electron properties microscopy revealed aggregated particles with a comparatively smooth surface. Different functional groups were identified by Fourier transformer Infrared spectroscopy including carboxylic groups, N-H, O-H, C-C, C-O-C and C-O-H bonds. Thermo-gravimetric analysis revealed three major regions of weight loss in the powder sample. The dielectric analysis showed that the powder had a dielectric constant of 171 at 95°C and a loss factor of 2.8. Overall, the study provided insightful data about the properties of freeze-dried watermelon rind powder, which could be useful for its application in the food industry.

Key words: Watermelon rind powder, functional properties, dielectric properties, surface morphology, X-ray diffraction

INTRODUCTION

Agro-industrial residues pose problems on the economic, social and environmental levels, prompting growing concern. The food processing industry, which includes processing fruits and vegetables, is the second largest waste producer in the world after domestic sewage (Sagar *et al.*, 2018). Given that by-products of fruits and vegetables are abundant with bioactive compounds, consumers' shifting views towards including natural bioactive compounds in the diet are the driving force behind the conversion of food waste into marketable products. One of the most popular fruits consumed worldwide is the watermelon (*Citrullus lanatus* L.), which has an estimated global production of 117 million tonnes (FAOSTAT, 2021). Watermelon is a member of the Cucurbitaceae family and has high water content. This fruit is rich in citrulline (non-essential amino acid), polyphenolic compounds and vitamin C, and its peel, seeds and rind are considered to be solid wastes for animal feeding. Watermelon rind, on the other hand, has a variety of industrial, pharmacological and food applications. Furthermore, agro-industrial residues can be a source of pectin and can further be utilized for the preparation of marmalades, jams, sweets, preserves and pickles. Several studies have been conducted recently on the use of WMR powders in the formulation of several types of baking items, including bread (Badr *et al.*, 2018), cookies (Naknaen *et al.*, 2016) and noodles (Ho and Che Dahri, 2016).

Moreover, the high moisture content of watermelon rinds makes them very perishable and prone to degradation when they're in fresh state. Enzymatic activity and microbial growth can be slowed or inhibited to

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extend the shelf life of the rinds by lowering the water activity (a_w) and moisture content to safe levels. These can be obtained by drying watermelon rinds and grinding them into a powder. Different drying techniques are used in the food sector as hot air, vacuum, spray and freeze-drying. Among all, freeze-drying was adopted in the current study as it results in products of the highest quality in terms of the exceptional sustaining of flavour, nutrients, polyphenolic compounds and aroma as well as sensory and physical qualities (Ho *et al.*, 2017). However, the comprehensive nutritional information of any powder material is required before its incorporation into food items, as powder materials undergo a variety of processing procedures during product development. The physico-chemical, colour characteristics, dielectric, functional and flow properties of the powder material will play a role in the method, the stability and the calibre of the food item into which it is inserted. Using thermo-gravimetric studies of food material, one can also determine at what temperatures and when drying is complete, as well as when the thermal decomposition of the food will begin. For the structural studies of fruit powders, scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transformed infrared spectroscopy (FTIR), techniques can be used. Various food products have been heated using radio frequency technology, commonly referred to as dielectric heating. For semi-solid and solid foods with limited thermal conductivity, radio frequency heating has a variety of advantages over other conventional treatment methods, including better quality, speedier heating, more even dispersion of heat and increased energy effectiveness. Dielectric properties of food materials are the most significant variables influencing radio frequency heating because they have a direct impact on food's ability to absorb and convert energy, which in turn affects heating rates and uniformity. The current investigation was aimed at ascertaining the physico-chemical, colour, dielectric, thermal and structural attributes of freeze-dried watermelon rind powder.

MATERIALS AND METHODS

Watermelon fruits (*Citrullus lanatus*) were procured from the local market in Hisar, Haryana, India. Fruits were washed, peeled and the rind was manually removed from the fruit, sliced into pieces, and then grounded into a paste. The prepared paste was pre-treated and frozen at -50°C overnight (12 h) and then freeze-dried at -80°C (Christ Alpha 1-2 LDplus). The dried watermelon rind was grounded into powder, sieved through 100 mesh size, packed and stored under refrigerated conditions until analysis (WRP: Ho and Che Dahri, 2016).

Nutritional components of prepared WRP included the sample's moisture content, which was determined using an oven drying technique. A conventional procedure was used to determine the amount of ash present. WRP samples were heated to a maximum temperature of 525°C in a muffle furnace for 6 h. The ash was then cooled in a desiccator and weighed. The Kjeldahl method was used to determine the protein content. The fat content was determined using the Soxhlet technique. Ranganna's methodology was used to calculate the amount of fibre. The difference between the sample's total weight and the total of its other components (crude fat, crude protein, moisture and ash content) was used to compute the sample's total carbohydrate content. The energy value of the WRP was calculated by multiplying the amounts of fat, protein and carbohydrates by the corresponding factorial values:

> Energy value (kcal/100 g) = [(carbohydrate % X 4) + (crude protein % X 4) + (crude fat % X 91)]

All proximate analyses were conducted in triplicate.

A chroma metre (HunterLab, ColorFlex EZ, Reston, VA, USA) was used to analyze the WRP's colour using the L*, a* and b* values, where, L* stands for lightness (0 = black, 100 = white), a* for red/green (+ value = redness, - value = greenness), and b* for yellow/blue (+ value = yellowness, value = blueness). According to the formulas hue = $\tan^{-1}$ (b/a) and C = $(a^2 + b^2)^{½}$, the hue angle and chroma (C) were computed. Estimates of the cohesion and flowability of powders were provided by analysis of flow characteristics (Vivek *et al.*, 2020). The bulk density (tapped and loose), true density, Carr index (CI) and Hausner ratio (HR), of the WRP sample were determined following Meghwal and Goswami (2017) and porosity (Vivek *et al.*, 2020). The HR and CI were estimated as:

Hausmer ratio (HR) = Tapped bulk density/Loose bulk density

Carr's index (CI) % = 100 X [(Tapped bulk density - Loose bulk density) / (Total bulk density)]

Functional traits of WRP were identified by analyzing its solubility, oil absorption capability, swelling and foaming capacity (Jha *et al.*, 2021).

FTIR spectrum for the WRP sample was measured by a Fourier transform spectrophotometer (PerkinElmer Spectrum Version 10.4.00). A manual tablet presser was used to crush the samples into pellets after gently combining them with micronized KBr powder. Measurements were done in the 4000-400/cm wavelength region at room temperature (25°C).

The morphological observations of WRP were done using high-resolution field emission scanning electron microscope (FE-SEM) with EDS (JSM-7601FPlus). The samples were gold coated to enhance their interface conductivity. The samples' digital photos were captured at a 10kV accelerating voltage.

The XRD analysis of the WRP samples was carried out using the PANalyticalXpert Pro XRD instrument. The investigation was conducted using Cu-K radiation at 1.540 Å as the source. The spectra were scanned with a diffraction angle (2??) in range of 5-80° and a step size of 0.03°/step and 0.6 sec/step.

TGA studies for WRP samples were performed with PerkinElmer Simultaneous Thermal Analyzer (STA) 6000 with a temperature range from 30 to 950°C and a heating rate of 15°C/min. The weight loss of fruit powder at various temperatures was determined in TGA analysis. The measurements offered quantitative and qualitative data about physical and chemical alterations involving endothermic or exothermic processes, as well as alterations in heat capacity.

A parallel plate capacitor technique was applied to ascertain WRP's dielectric properties WRP (Mehra *et al.*, 2022). An LCR metre (HIOKI, IM3536) with two probes was employed to assess the dielectric characteristics, between the temperature range from 20 to 160°C with a frequency range from 1 Hz to 1.5 MHz. WRP pellets (2.15 mm in thickness and 6 mm in diameter) were made using a KBR hydraulic press. To make the manufactured WRP pellets conductive and adhere to copper wire, to help the device function as a parallel plate capacitor, a silver paste was applied to both sides of the pellets.

All the observations used in the above study were collected in triplicates and shown as Mean±SD. The 2019 version of Microsoft Excel was used to evaluate the coefficient of variation (CV; Microsoft Corporation, Microsoft Way Redmond, USA).

RESULTS AND DISCUSSION

The current work examined the physical, chemical, functional, thermal, dielectric and surface properties of freeze-dried watermelon rind powder. When creating formulae for novel food products, understanding the chemical composition of the powder is essential. The nutritional composition of WRP has been listed in Table 1. The findings of the proximate composition of WRP revealed that it had 10.22 ± 0.17% moisture content. The moisture content of any powder sample significantly influenced the rate of reconstitution and the physical state of a sample. The low moisture content of WRP facilitated its long-term sustainability and additional product development integration. In relation to the relative protein, and ash levels of WRP, the sample had 11.16 ± 0.19% protein content and 12.93±0.11% ash content. Any powdered product's solubility capacity or dissolution rate was significantly influenced by its fat content. The level of fat content obtained in WRP sample was 1.30±0.12%. Also, the low-fat content obtained in studied WRP samples reduced the chance of rancidification and oxidation of the powder by extending its shelf life. The total

Table 1. Descriptive statistics of nutritional analysis of WRP

Parameters	Range	Mean values	Standard deviation
Moisture content (%)	10.05-10.45	10.22	0.17
Ash content (%)	12.79-13.06	12.93	0.11
Protein content (%)	10.9-11.34	11.16	0.19
Fat content (%)	1.15-1.45	1.30	0.12
Carbohydrates (%)	63.16-63.59	63.38	0.18
Crude fibre (%)	16.48-16.97	16.74	0.20
Water activity (aw)	0.40-0.404	0.402	0.002
Energy (kcal/100 g)	311.89-313.46	312.45	0.72

carbohydrate content of WRP was 63.38 ± 0.18%. Proximate composition of WRP, when compared to earlier reported values was similar, except for higher values for fat (2.38%) and carbohydrate content (73.18%). WRP had higher values of fat, moisture, protein and ash content in comparison to the results reported by Naknaen *et al.* (2016), while they were in accordance with Chakrabarty *et al.* (2020). The findings of the study showed that the total dietary fiber content of the WRP ranged from 16.48-16.97%. The WRP's fibre content suggests that it may be used as an enrichment agent in foods to make high-fiber foods, and nutrient-rich diets with added value. The fiber content of WRP was higher in comparison with the findings of 12.78 and 15.98% (Chakrabarty *et al.*, 2020) and compared to the stated values (21.24%) was lower than Shivapour *et al.* (2020). The insoluble fiber in WRP was associated with several health advantages, among them was a decreased risk of cardiovascular diseases and the prevention of diabetes, weight gain and several types of cancer. When evaluating the energy of WRP, it was depicted that the sample had 312.45 ± 0.72 kcal/100 g. The results of energy obtained in case of WRP were higher than the findings of 295.78±0.48 kcal/100 g (Badr *et al.*, 2018); indicating the water activity in food systems for biochemical processes and microbiological activity is a crucial metric. Any food product that has a water activity value of b < 0.6 is microbiologically stable and does not exhibit any growth of spoilage or pathogenic organisms. Regarding the WRP's water activity the powder showed values ranging from 0.402±0.002. WRP's water activity depicted in the study was lower than 0.6. Based on the results of nutritional analysis, the dried WRP appeared as functional constituents with a potential for excellent storage stability and microbiological security.

The powder's colour is one of the qualities that most clearly identify it as a natural colourant. Table 2 lists the values of the colorimetric analysis's parameters, including luminosity (L*), a*, b*, chroma*, and hue values. 53.72 L* value was displayed by the WRP sample, showing a tendency towards whiteness in colour. The rind's green colouring and the presence of a pigment chlorophyll were indicative of the WRP sample's negative value (-0.67) for *a**. The WRP samples attained the value of 21.04, and the yellowish tint served as a visual cue for the positive value of *b**. The hue angle (-88.17) was negative for the WRP samples because the values of *a** were negative. The WRP sample's chroma value was 21.05. The final product was stated to have a slight green tint (Fig. 1).

Fig. 1. Colour properties of freeze-dried watermelon rind powder (WRP); a = red (+) - green (-) colour and b = yellow (+) - blue (-) colour; Hue = tan-1 (b/a) and Chroma = $(a2 + b2)^{½}$.

Powdered material's flow ability can be determined using the Carr index and Hausner ratio. The powder's physical characteristics, such as surface structure, shape, size, bulk density and particle density, typically influence flow ability. The bulk density of the powder, which is determined by characteristics including inter particle forces, particle size and the strength of contact points, is a crucial determinant of the convenience of transporting and packaging particulate food. The bulk density of WRP was calculated as 0.3211±0.006 g/cm^3. A powder sample's tapped density, which allows for maximum packaging of the sample under the impact of outside pressures, is one of its key quality characteristics. Tapped bulk density of WRP was 0.4103±0.007 g/cm^3. The

Table 2. Colour characteristic of watermelon rind powder (WRP) in three replications

Sample	L*	a*	b*	Hue angle (H)	Chroma (C)
WRP	53.72±021	-0.67±0.09	21.04±0.15	-88.17±0.26	21.05±0.15

processing and transportation of powders require certain important flow properties including the Carr's index (CI) and Hausner ratio (HR). While HR calculates the inter particulate friction, CI assesses the stability of the powder and the strength of the bridge (Vivek *et al.*, 2020). The HR and CI of WRP were 1.27±0.04 and 0.21±0.02%, respectively. For better powder flow qualities, lower CI and HR values are recommended. The ability of a powder to dissolve in water is an extremely important determining element. It displays the characteristics of powder samples, including their digestibility. For samples of WRP, the water solubility index ranged from 54.125 to 55.987%. The swelling power of WRP ranged from 6.9635 to 7.4523 ml/g DW. The particle size, hydro-phobicity of the powder, overall charge density, chemical make-up and drying techniques all affect oil absorption capacity (OAC). OAC is crucial for flavour retention and offers food products like baked goods a spongy feel. The OAC of WRP was in the range of 2.04 to 2.49 g/g. Processing techniques, pH level and surface tension typically have an impact on foaming capacity. The foaming capacity of the WRP sample ranged from 14.15 to 14.86%. Fig. 2 displays the results of an FTIR examination of WRP. Below 900 is the last spectral region known as the "fingerprint" (Ramírez-Hernández *et al.*, 2019) to identify conformational changes and crystal regions in the material under study. The region of the infrared spectrum where each organic chemical has its distinct line of absorption is the region of the fingerprint. Those lines provide details on the presence of different functional groups that exist in the sample being studied (Ramírez-Hernández et al., 2019). C-Br stretch alkyl halides are attributed to peaks in the 650-510/cm range, with a peak at 622.99/cm in the WRP sample. The peak at 777.67/cm in WRP can be attributed to (ethyl branch ethylene butane). The sample's peak at 818/cm may be associated with CH_3 rocking or N-H broad loop bending. Strong expanding bonds between distinct oligo- and polysaccharides' C-O, C-C, C-O-C, and C-O-H are indicated in the spectrum areas between 1500 and 900/cm. Different peaks in this region viz., 917.27, 1057.47, 1238.35 and 1384.74/cm, reflected the presence of carbohydrates in WRP. N-H absorption may be the cause of the peak at 1633.85/cm. The bands at 1743/cm may represent free and esterified carboxylic groups, which may help to identify pectin (Husein *et al.*, 2017). A peak near 2925/cm, which is detected in the WRP sample, can be because of methylene's asymmetric aliphatic, C-H stretching vibrations (–CH_2). As broad absorption was observed at 3435/cm, this is attributed to vibrations of -OH stretching (Husein *et al.*, 2017).

The surface characteristics of a powder can be determined from its SEM results, which can be helpful for a variety of applications, such as determining the particle size and shape for

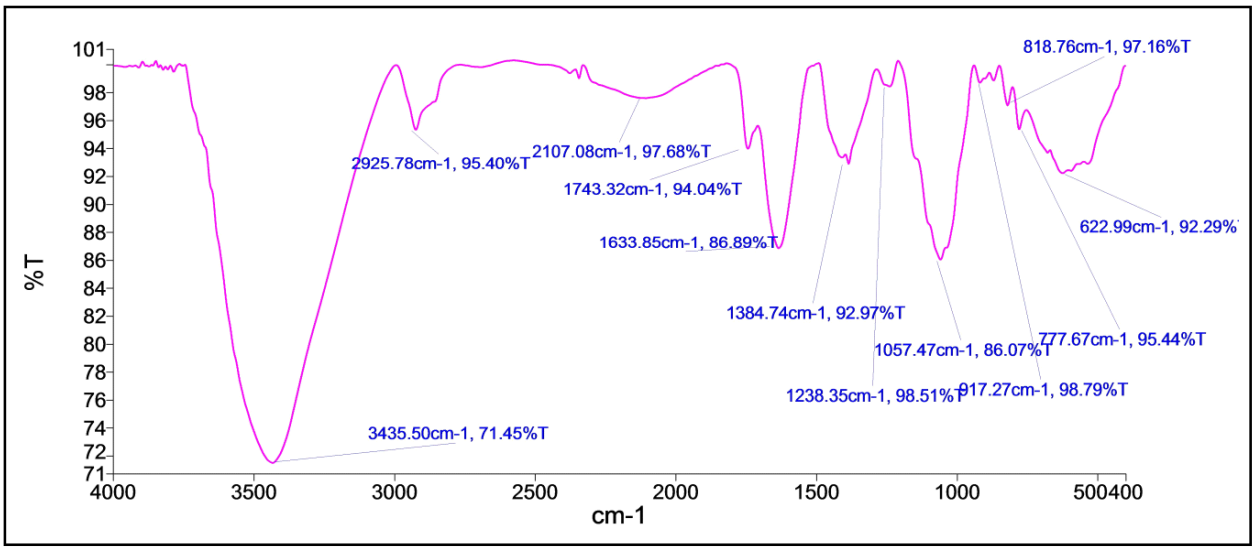
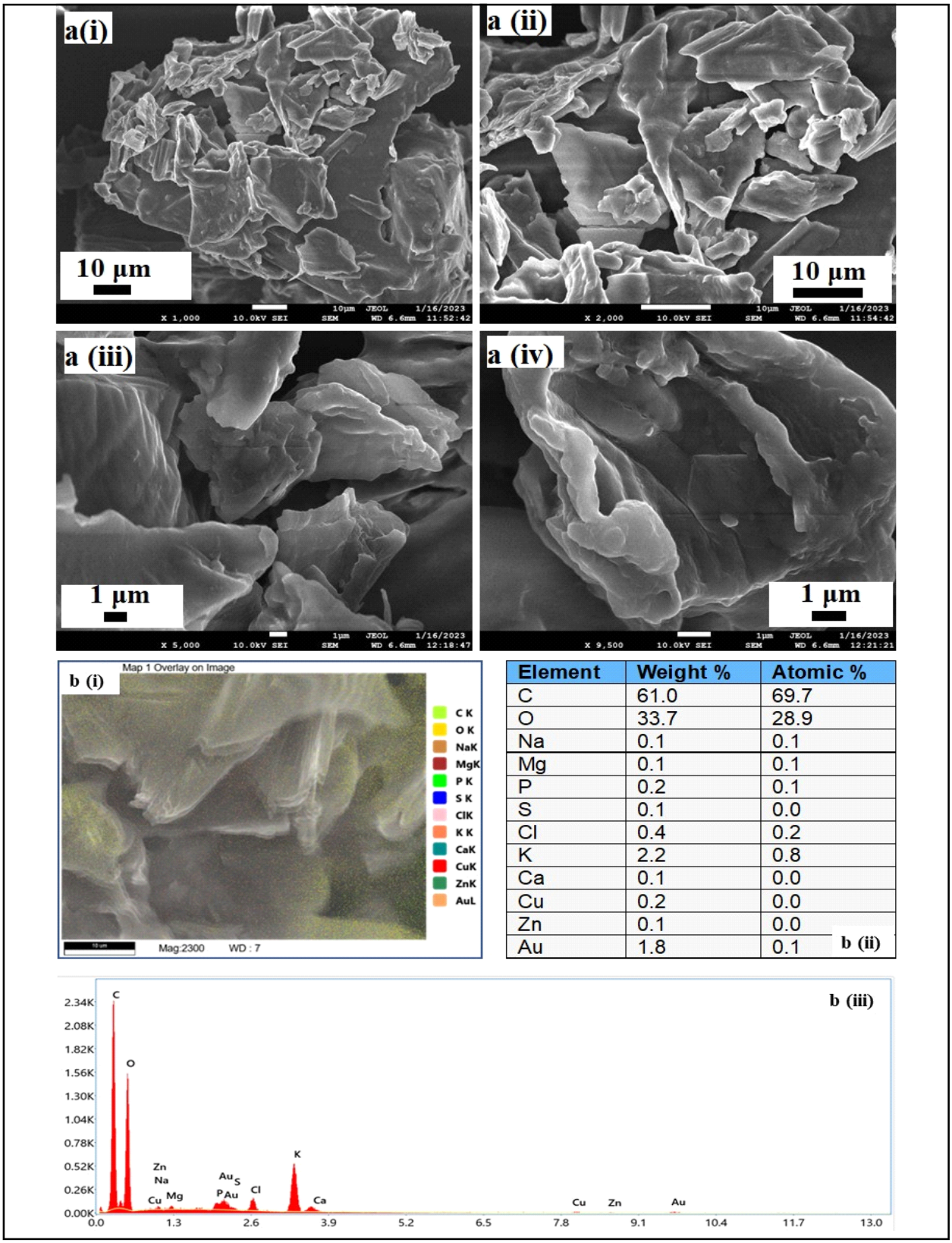


Fig. 2. FTIR spectra of watermelon rind powder (WRP).



Element	Weight %	Atomic %
C	61.0	69.7
O	33.7	28.9
Na	0.1	0.1
Mg	0.1	0.1
P	0.2	0.1
S	0.1	0.0
Cl	0.4	0.2
K	2.2	0.8
Ca	0.1	0.0
Cu	0.2	0.0
Zn	0.1	0.0
Au	1.8	0.1

Fig. 3. (A) SEM micrographs of watermelon rind powder (WRP) at different magnifications; a (i) 1000x, a (ii) 2000x, a (iii) 5000x, a (iv) 9500x and (B) Elemental mapping of WRP; b (i) Elemental distribution (dot) map, [b (ii) and b (iii)] EDS spectrum.

formulation needs or comprehending the surface topography and chemistry for functionalization needs. Fig. 3 (a) displays the SEM micrograph of WRP at various magnifications. The obtained SEM images of WRP revealed amorphous particles with a comparatively smooth surface. Partial aggregation of small particles to larger ones can also be seen. The elemental distribution on the surface of WRP is shown in Fig. 3b (i), surface's minerals and compositional elements (C, O, Mg, Na, P, Cl, S, K, Cu, Ca, Zn and Au) can be seen uniformly distributed. Additionally, Fig. 3b (ii) displays the EDS elemental mapping of each element. Further, Energy-dispersive X-ray spectroscopy (EDS) was used to examine the elemental makeup and distribution of WRP. In addition to the compositional elements oxygen and carbon, the EDS spectrum in Fig. 3b (iii) revealed the presence of chlorides, potassium, phosphorus, copper, sodium, calcium, magnesium, zinc and sulphur on the surface of WRP.

Fig. 4 depicts the X-ray diffraction patterns of WRP. The XRD results of watermelon rind powder were analyzed to determine the structure of the sample. The XRD pattern showed various distinct peaks in a broad region at 2θ values of 16.8°, 22.6°, 20.1, 20.7, 21.6 and, which were identified as the diffraction peaks of hemi-cellulose, pectin and crystalline natural cellulose (cellulose I) in agricultural biomass, respectively (Wang *et al.*, 2022).

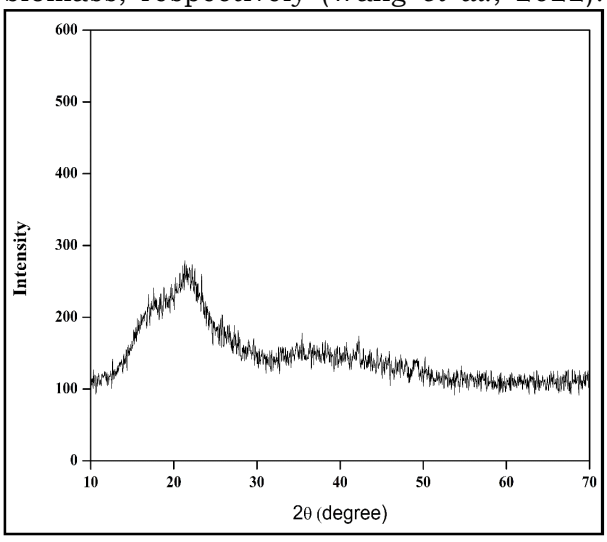


Fig. 4. X-ray diffraction patterns of watermelon rind powder (WRP).

These results indicate that the watermelon rind powder is primarily composed of polysaccharides, which are commonly found in plant-based materials. The broad reflections demonstrated WRP having an amorphous structure. In contrast to crystalline powders, which may require extended periods of time and/or specific conditions to dissolve completely, amorphous powders can dissolve rapidly in a variety of solvents, including water. This property makes amorphous powders ideal for use in various food and beverage applications, where rapid dissolution is desirable.

The TGA analysis revealed thermal stability and decomposition behaviour of WRP. The TGA curve showed a gradual weight loss with increasing temperature, indicating the presence of both volatile and non-volatile components in WRP. This information can be used to optimize processing conditions, such as drying temperature and time, to preserve the quality and composition of the powder. Based on the results of TGA, WRP typically followed two main weight loss phases: The drying phase, which was the initial stage of weight loss and corresponded to the sample's moisture being extracted. The weight loss during this phase depends on the sample's initial moisture content. The degradation phase is the second stage of weight loss and corresponds to the decomposition of organic matter in the sample. TGA was used to examine the thermal stability of freeze-dried WRP. In Fig. 5, the TGA curve for WRP was displayed. Three major areas of weight loss may be seen on the TGA curves of guava powder (28.68-106.88, 106.88-260.79 and 260.79-366.36°C). A weight drop of about 5.96% was visible in the first section (28.68-106.88°C). Water loss during the temperature increase can be believed to be the cause of this mass loss. In the second indicated region (106.88-260.79°C), the WRP had a twice-stage mass reduction (31.86%), which was likely due to the more complicated volatilization of materials. Demethoxylation, dehydroxylation and decarboxylation reactions must be taking place on pectin and related polysaccharides between 150 and 350°C. After the volatilization of water and other substances, TGA curve analyses revealed a more stable state in the

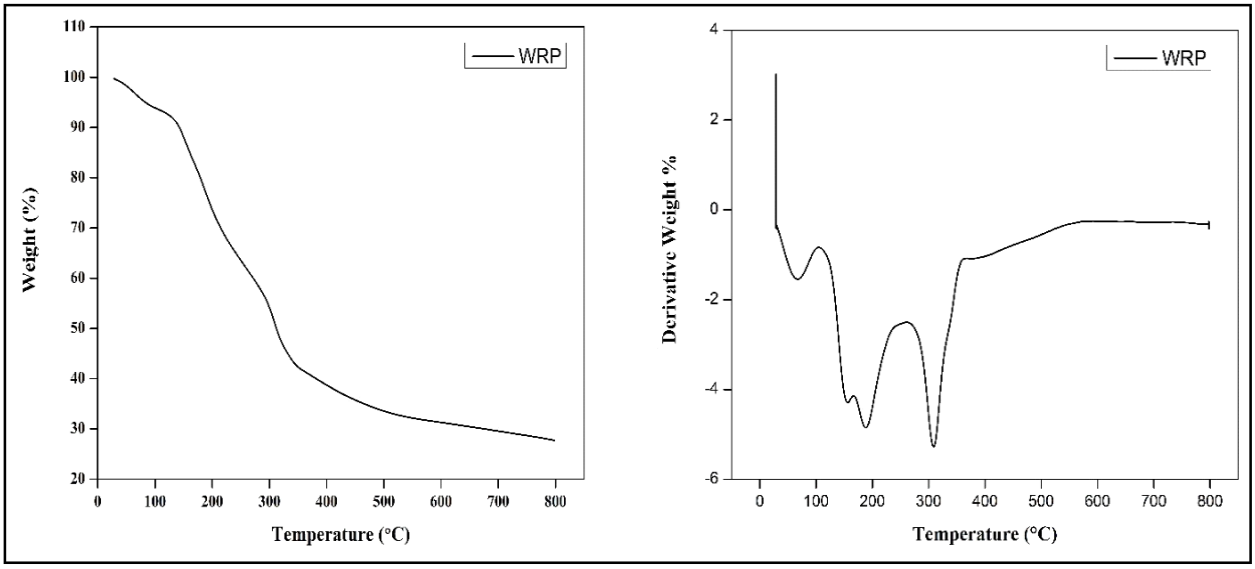


Fig. 5. (a) TGA curve of watermelon rind powder (WRP) and (b) TGA derivative weight curve of watermelon rind powder (WRP).

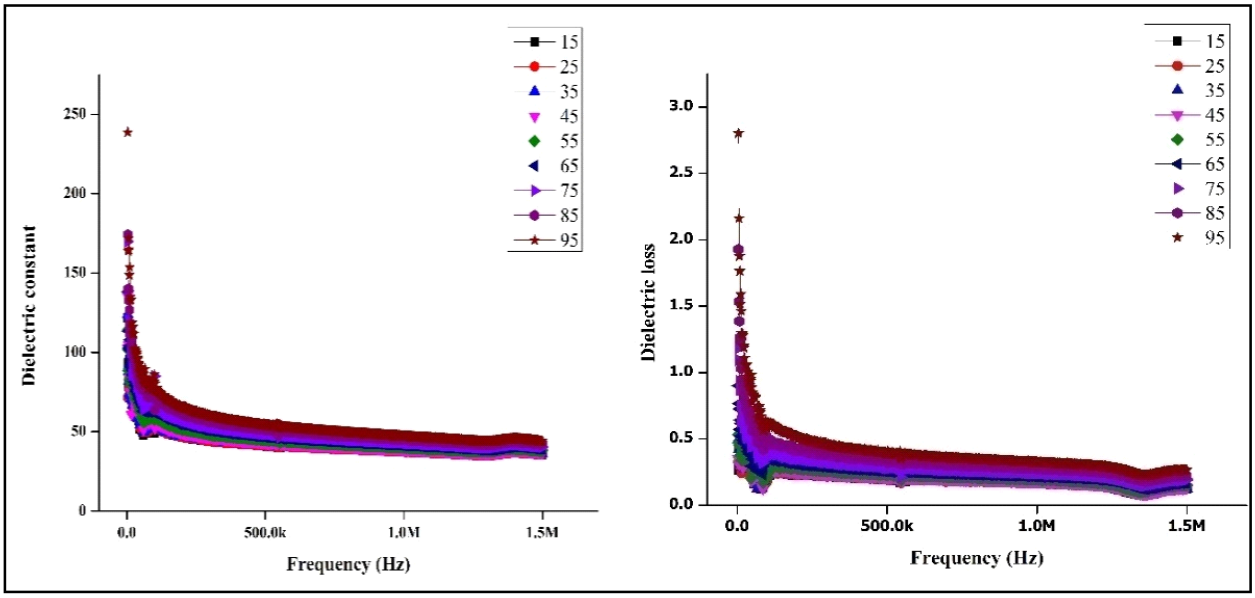


Fig. 6. Dielectric properties of watermelon rind powder (WRP).

third region (260.79-366.36°C). The weight that was still intact after the incineration was 27.62%, demonstrating the WRP's thermal stability.

The dielectric constant is a function of frequency for a temperature range of 15 to 95°C. The dielectric constant of WRP exhibited an increasing trend with temperature increase, reaching a maximum of 171 at 95°C, and exhibited a decrementing tendency with increasing frequency at all temperatures. The values dropped significantly from 0.001 to 0.2 MHz (Fig. 6). The dielectric loss factors for WRP were displayed against frequency in a temperature range ranging from 15 to 95°C. Similar to the dielectric constant, WRP demonstrated a sharp fall in the dielectric loss factor with increasing frequency in the initial phase which was gradually decreasing until a notable decrease around 1.3 MHz. This quick decrease was followed by an increase in the dielectric loss value. The dissipation of energy at high temperatures was confirmed by the increase in dielectric loss with temperature (Mehra *et al.*, 2022). At 95°C, the dielectric loss reached its greatest value (2.8), which

decreased to 0.56 at 0.08 MHz. Elevated electric polarisation at higher temperatures may be related to the rapid rise in dielectric loss values with temperature. Several authors have noted changes in dielectric properties of materials as a function of temperature and frequency (Lin *et al.*, 2016; Dag *et al.*, 2019), which may be related to water dipole activity, increased bound water mobility, compositional element stability, or reduced ionic conductivity. The frequent drop in dielectric constant and loss factor in WRP may be due to changes in ionic conduction and ion mobility in the sample. Ionic conduction predominates the mechanism of dielectric heating below 300 MHz, and as frequency increases, ionic conduction diminishes, resulting in a fall in the value of dielectric loss. Various samples of low-moisture foods, including wheat flour and lentil, green pea, chickpea and soybean flour displayed patterns for the dielectric constant and loss factor that was similar.

CONCLUSION

The physicochemical, dielectric, functional, thermal, structural and flow characteristics of freeze-dried watermelon rind powder demonstrated its potential applications. The powder exhibited the desirable physico-chemical properties, also maintaining its structural and functional properties during the freeze-drying process. X-ray diffraction demonstrated fruit powders' semi-crystalline profile and average particle size. Scanning electron microscopy revealed aggregated particles with a comparatively smooth surface. Different functional groups were identified in WRP using FTIR. WRP exhibited good thermal properties analyzed by TGA. The dielectric properties of the powder showed its potential for microwave assisted processing, and the flow characteristics indicated its potential for use in various food processing operations. The findings of this study demonstrate the need for a paradigm shift to take watermelon rind powder into account from new management perspectives, where the potential of the rind becomes a crucial factor in how this agro-industrial residue is utilized. Additional focused studies should be started to develop fresh frameworks for the use of watermelon rind in agronomic and environmental balance and the development of value-added products in the food and non-food industries.

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