

# Bioactive Molecules in Food: from Food Composition and Dedicated Databases to Metabolomic Pathways

Special Issue Editor in Chief: Alessandra Durazzo

Guest Editors: Massimo Lucarini and Márcio Carochó



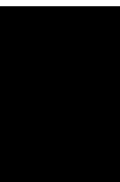


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## Editorial

# Bioactive Molecules in Food: From Food Composition and Dedicated Databases to Metabolomic Pathways

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The categorization and classification of compounds with nutritional and nutraceutical features have emerged as a need for consumers and industry, and the development and implementation of specific and dedicated databases throughout harmonized and standardized approaches based on both analytical and collected data taken from the literature are crucial. Food Composition Databases and dedicated databases are addressed to produce, collect, and present data in a standardized format to compare data from different databases and countries.

The construction of databases is based on standardized and harmonized procedures for production, collection, compilation, and publication of food data (description, selection, collection, preparation, references, analytical approach and/or value documentation, calculation, and compilation). The design and construction of food databases require foremost the exact identification of foods through an adequate food nomenclature and a precise description. A coherent food description and classification system is essential. In addition, the understanding of the activities and benefits of biologically active compounds in humans is crucial. Updated information on bioaccessibility, bioavailability, and pharmacokinetics of target compounds, new data on novel dietary biomarkers, and an assessment of metabolites pathways are needed.

This special issue explored the following topics: (i) distribution and occurrence of compounds with nutrient and nutraceutical attributes in foods and food groups; (ii) original analytical data, data from the literature, and data

traceability; (iii) plant metabolic pathway databases; (iv) new datasets/dedicated databases for nutrients and bioactive compounds; (v) metabolite data and new sets of biomarkers; (vi) aspects of data structure and data mining from currently available and published data and how to include or exclude them; (vii) compositional data on new foods and food products available in the market; (viii) optimization of sample description and sampling procedures as well as value documentation and calculation procedures, i.e. recipe calculation, assessing yield and retention factors, performing quality data evaluation index; (ix) datasets/databases on conventional and emerging categories of foods: traditional, certified, branded foods and recipes, as well as functional and fortified foods, non-conventional foods, and food waste; (x) classification of food groups, with focus on emerging categories and ontologies and application of description and classification food systems, examples of coding procedures, and semi-automatic and automatic systems; (xi) matching processes for linking food composition data to food consumption data; (xii) food labeling.

In this context, Sagar et al. [1] described and gave new data on physicochemical and thermal characteristics of onion skin from fifteen Indian cultivars for possible food applications. Another example given by Kumar et al. [2] evaluated the chemical, functional, spectral, and thermal characteristics of *Sargassum wightii* and *Ulva rigida* from the Indian Coast.

All articles, which are part of this Special Issue, reflect modern trends and outline new ideas for future network

collaborative research from the perspective of interoperability and sharing data for development, management, applications, and benefits of Food Composition Databases and Dedicated Databases, by contributing to the growth of this area of research and adding information scientifically substantiated by new data.

We hope that the readers will find this Special Issue interesting and inspiring.

### **Conflicts of Interest**

The Guest Editors declare that there are no conflicts of interest.

### **Acknowledgments**

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## Research Article

# Technological, Sensory, and Hypoglycemic Effects of Quinoa Flour Incorporation into Biscuits

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**Background.** Biscuits are consumed by all of society in the world. Incorporation of different ratios of quinoa flour into wheat flour for the production of biscuits is needed for the production of functional foods. **Objective.** This study aimed to evaluate the incorporation of 12.5% or 25% quinoa flour into biscuit production, evaluate rheological and sensory characteristics, and investigate the effect of the consumption of 20% cooked biscuits on diabetic rats. **Design.** The gross chemical composition, total carotenoids, phenolic and flavonoids of wheat flour and quinoa flour, and the rheological properties of the control, 12.5% quinoa, and 25% quinoa biscuit dough were determined. The effects of consumption of 12.5% quinoa and 25% quinoa biscuits on diabetic rats were investigated. **Results.** Quinoa flour had significantly higher levels of the gross chemical composition except for carbohydrate and increased phenolic compound and flavonoids content than those in wheat flour. Increasing the amount of quinoa flour in the biscuits could increase the farinograph and extensograph values of the dough. Biological results showed that the highest improvement in nutritional values appeared in the diabetic rat group, which consumed 25% quinoa biscuit for 60 days. The consumption of 12.5% quinoa biscuit and 25% quinoa biscuit showed a decline in blood glycosylated hemoglobin and glucose and an elevation in insulin levels compared with the positive control diabetic rat group. **Discussion and Conclusion.** It is encouraging to replace wheat flour with quinoa flour in biscuit manufacturing owing to positive effects on both the technological properties and sensory evaluation of biscuits. The increase of quinoa flour up to 25% had favorable nutritional values and hypoglycemic effects.

## 1. Introduction

Quinoa (*Chenopodium quinoa* Willd.) is a member of the Chenopodiaceae family and acts as pseudocereal due to yield grains and consumed as cereals or flour products [1, 2]. Quinoa has high nutritional value and acts as a complete food. It offers all essential amino acids, such as methionine, histidine, and lysine; essential fatty acids, such as oleic and linoleic acids; and dietary fiber, which detoxify the waste products and toxins. It also has a wide range of minerals and vitamins, more than those present in cereals and legumes [3–7].

Quinoa has been cultivated worldwide as a new crop species directed at technological and commercial

importance. It is used in cooking, baking, modified food products such as breakfast cereals, pasta, and cookies, industrial use of starch, protein, and saponin, and the pharmaceutical industry and animal feeding [1, 8–10].

Currently, quinoa is incorporated into wheat flour at a definite percentage for bakery production as cakes, bread, and biscuits to enrich the sensory characteristics of the baked goods, such as the aroma, taste, texture, and acceptability, in addition to increasing the nutritious and health benefits of baked products [11–13]. Quinoa is known as a functional food because it improves lipid parameters, triglycerides, and the glycemic index in addition to its beneficial components, such as vitamins and minerals, and is gluten free [7, 14].

Biscuits are consumed by nearly all of society because of their low cost, availability, taste variation, and relatively long shelf life in addition to their good nutritional quality, which leads to the importance of increasing their protein, vitamin, and fiber content. It has been of interest to improve the nutritional and sensory quality of biscuits by incorporating functional foods, such as those in gluten-free biscuits. A 60% substitution of wheat flour with quinoa flour resulted in dark-colored cookies with increased hardness and decreased volume and chewiness, so the addition of quinoa flour should be not more than 50% to enhance nutritional and sensory properties [15–19].

Therefore, this study aimed to determine the incorporation of different ratios of quinoa flour into wheat flour for the production of biscuits, to evaluate the rheological and sensory characteristics, and investigate the effect of the consumption of 20% biscuits on diabetic rats.

## 2. Materials and Methods

Wheat flour, quinoa flour, sugar, salt, eggs, margarine, lecithin, baking powder, and vanillin were purchased from local markets. Twenty-eight albino rats weighing  $120 \pm 7$  g were provided by the experimental animals' center in the Research Center in Prince Sultan Military Medical City, Riyadh. Ethical guidelines for investigations involving experimental animals were followed throughout the study. The experiments were carried out with the help of the staff of the Scientific Research Center of the MSD at their experimental animal (REC AP2411/16A). The basal diet was formed [20].

The gross chemical composition of wheat flour and quinoa flour, including protein, lipids, fiber, ash, and moisture, was estimated according to AACC methods [21]. The carbohydrate content (%) of wheat flour and quinoa flour was calculated.

The total phenolic compound content of wheat flour and quinoa flour was determined calorimetrically and expressed as gallic acid equivalents (gGAE/1000 g) [22]. The total carotenoid and flavonoid contents were determined spectrophotometrically according to standard methods [23]. The water-holding capacity and oil-holding capacity of wheat flour and quinoa flour were determined [24, 25].

The ingredients of different biscuit batters illustrated in Table 1 were mixed, rested for half an hour, laminated, shaped, and then, baked at  $220^{\circ}\text{C}$  for 15 min to prepare control, 12.5% quinoa representing 6.22% of dry ingredients of biscuit, and 25% quinoa biscuits representing 12.44% of dry ingredients of biscuit [26].

The rheological properties of the control, 12.5% quinoa, and 25% quinoa biscuit dough were determined by using Brabender farinograph and extensograph instruments according to the AACC method [21]. Sensory evaluation of the control, 12.5% quinoa, and 25% quinoa biscuits was conducted for appearance, texture, color, taste, odor, and overall acceptability by ten trained sensory panelists using a hedonic scale.

To investigate the antidiabetic effect, rats fed a basal diet and water *ad libitum* were allowed to adapt for one week. Rats were injected intraperitoneally with streptozotocin at a

dose of 50 mg/kg body weight in 0.1 M citrate buffer to induce diabetes, which was confirmed by the elevation of blood glucose samples on the third day. Rats were randomly divided into four groups as follows:

The positive control rat group consumed a basal diet.

The control biscuit rat group consumed the basal diet containing 20% dried, powdered control biscuit.

The 12.5% quinoa biscuit rat group consumed the basal diet containing 20% dried, powdered 12.5% quinoa biscuit.

The 25% quinoa biscuit rat group consumed the basal diet containing 20% dried, powdered 25% quinoa biscuit.

Daily food intake and weekly body weight were recorded. The feed efficiency ratio (FER) was calculated.

Rats were sacrificed after 60 days to estimate hemoglobin, glycosylated hemoglobin, glucose, and insulin [27]. The obtained results of triplicate assays were statistically analyzed by analysis of variance (ANOVA), and significant differences among Duncan's multiple tests were determined using SPSS computer software.

## 3. Results and Discussion

The analysis of the wheat flour and quinoa flour samples used in the production of the control, 12.5% quinoa, and 25% quinoa biscuits is presented in Table 2. The wheat flour had significantly lower protein, lipids, fiber, ash, and energy levels than quinoa flour. On the other hand, wheat flour had higher contents of carbohydrate and moisture than quinoa flour. The results were in agreement with the results of many authors [5, 11, 28, 29]. In contrast, quinoa flour had 58.3% carbohydrate, 13.5% crude protein, 9.5% crude fiber, 1.2% total ash, and 11.2% moisture. Also, 12% protein, 6.5% lipid, 77% carbohydrate, and 3% mineral contents in quinoa grain were estimated [30]. The variation in the chemical composition of quinoa flour was related to the area of cultivation and germination period [17].

Table 3 shows that higher total carotenoid, phenolic, and flavonoid contents were observed in quinoa flour than in wheat flour. These values were consistent with many authors who reported that quinoa has significant amounts of antioxidant compounds such as carotenoids, polyphenols, and phenolic acids [4, 25, 31]. Quinoa has kaempferol, ferulic, gallic acids, isorhamnetin, and routine bioactive components [4, 32]. Polyphenols, phytosterols, isoflavones, and flavonoids have excellent antioxidant properties and contribute nutraceutical benefits. The quinoa flavonoid level was higher than that of blueberries, reaching levels similar to those of flavonoid-rich fruits [33, 34]. The variations in mineral, saponin, and total phenolic compound contents in quinoa are mainly due to methods of processing and cooking [35].

Table 4 shows that the differences between the water-holding capacity and oil-holding capacity of wheat flour and quinoa flour showed a significant difference. Wheat flour had significantly higher water- and oil-holding capacity

TABLE 1: Ingredients in control, 12.5% quinoa, and 25% quinoa biscuits.

	Wheat flour (g)	Quinoa flour (g)	Margarine (g)	Sugar (g)	Egg (g)	Lecithin (g)	Vanillin (g)	Salts (g)	Baking powder (g)	Water (g)
Control biscuit	200	—	32	60	100	1.2	0.5	1	7.2	30
12.5% quinoa biscuit	175	25	32	60	100	1.2	0.5	1	7.2	30
25% quinoa biscuit	150	50	32	60	100	1.2	0.5	1	7.2	30

TABLE 2: Chemical composition of wheat flour and quinoa flour.

Nutrient flour	Protein (g/100 g)	Lipids (g/100 g)	Fiber (g/100 g)	Ash (g/100 g)	Carbohydrate (g/100 g)	Moisture (g/100 g)	Energy (kcal/100 g)
Wheat	10.99 ± 0.95b	1.45 ± 0.08b	1.75 ± 0.06b	1.69 ± 0.07b	76.13 ± 3.61a	7.99 ± 0.88b	361.53 ± 4.66b
Quinoa	15.68 ± 1.75a	6.64 ± 1.14a	3.40 ± 0.71a	4.55 ± 0.81a	63.13 ± 3.78b	6.60 ± 0.75a	379.02 ± 3.55a

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

TABLE 3: The total carotenoid, phenolic, and flavonoid content of wheat flour and quinoa flour.

	Total carotenoid (mg/100 g)	Total phenolic compound (mgGAE/100 g)	Total flavonoids (mg/100 g)
Wheat flour	0.10 ± 0.001b	30.18 ± 5.41b	11.17 ± 2.88b
Quinoa flour	1.45 ± 0.25a	66.45 ± 8.15a	32.14 ± 6.11a

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

TABLE 4: Water-holding capacity and oil-holding capacity of wheat flour and quinoa flour.

	Water-holding capacity (g H <sub>2</sub> O/g)	Oil-holding capacity (g oil/g)
Wheat flour	6.33 ± 0.22a	8.01 ± 0.66a
Quinoa flour	2.88 ± 0.07b	4.11 ± 0.65b

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

values than the values of quinoa flour. It is known that quinoa has expanded usages owing to its technological characteristics, such as solubility, gelation, water-holding capacity, and foaming, in addition to functional starch and omega-6, so it is appropriate to incorporate quinoa flour into wheat flour. The fiber of quinoa increased the water- and oil-holding capacities of the four mixtures, improving the dough and baked product quality [11, 30, 36].

The results in Table 5 show that the 12.5% quinoa biscuit and 25% quinoa biscuit had increased water absorption, arrival time, dough stability, and farinograph quality number values compared to the control biscuit. The highest farinograph parameters were observed in the 25% quinoa biscuits. These results were associated with hydrophilic carbohydrate, protein, and crude fiber contents in quinoa flour [28, 37–39]. In contrast, the obtained results disagreed with the decline in the arrival time, development, and stability of dough with increasing quinoa flour levels.

Increasing the quinoa flour in biscuit could increase extensibility, relative resistance, proportional number, and energy compared to the control biscuit. The values were highly significant in 25% quinoa biscuit. Also, relative resistance and proportional number were highly significant in 25% quinoa biscuit compared to the control biscuit, as represented in Table 6. The obtained results are correlated to

the dilution of an interrelated dough gluten matrix. In particular, biscuits have high proportions of fat and sugar and a short mixing phase, but the gluten network developed in the tested biscuits is related to an increase in the incorporation of the quinoa flour into the cookies and starch gelatinization [7, 40].

The data in Table 7 show nonsignificant differences in appearance, texture, color, and taste score and a significant lowered score of flavor and acceptability between 12.5% quinoa and control biscuits. The 25% quinoa biscuit had a significant decrease in the tested sensory characteristics compared with the control biscuit. The 25% quinoa biscuit showed significantly lower values of appearance, texture, color, and taste score and nonsignificant difference of flavor and acceptability compared to 12.5% quinoa biscuit. Generally, increasing the amount of quinoa flour in the biscuits affected their sensory evaluation due to the high levels of protein in quinoa flour, which affected the texture, flavor, and acceptability of the biscuits. These changes in sensory quality were attributed to the formation of hydrogen bonds among the hydroxyl carbonyl, amide hydroxyl groups, and polar groups of the other ingredients of the biscuit flour. Additionally, the increased protein content in quinoa flour formed tiny, compact bubbles in the dough structure, hence producing biscuit hardness [41, 42]. On the other hand, the

TABLE 5: Farinograph parameters of control biscuit, 12.5% quinoa biscuit, and 25% quinoa biscuit.

	Water absorption (%)	Arrival time (min)	Dough development time (min)	Dough stability (min)	Farinograph quality number
Control biscuit	56.61 ± 1.41c	1.30 ± 0.11c	3.01 ± 0.22b	4.32 ± 0.40b	132 ± 3.41c
12.5% quinoa biscuit	62.55 ± 2.14ab	2.23 ± 0.18b	4.31 ± 0.59ab	6.98 ± 1.11a	156 ± 4.98b
25% quinoa biscuit	67.01 ± 3.11a	3.55 ± 0.44a	5.03 ± 0.65a	7.35 ± 1.44a	167 ± 6.35a

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

TABLE 6: Extensograph parameters of control biscuit, 12.5% quinoa biscuit, and 25% quinoa biscuit.

	Extensibility (mm)	Relative resistance (BU)	Proportional number	Energy (cm <sup>2</sup> )
Control biscuit	163 ± 5.51c	490 ± 10.15c	2.75 ± 0.23b	108 ± 6.77c
12.5% quinoa biscuit	182 ± 4.57b	538 ± 8.61ab	3.41 ± 0.66a	119 ± 5.66b
25% quinoa biscuit	193 ± 2.15a	550 ± 6.71a	3.01 ± 0.44a	127 ± 2.11a

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

TABLE 7: Sensory evaluation of control biscuit, 12.5% quinoa biscuit, and 25% quinoa biscuit.

	Appearance	Texture	Color	Taste	Flavor	Acceptability
Control biscuit	8.10 ± 0.10a	8.10 ± 0.10a	8.20 ± 0.13a	8.24 ± 0.03a	8.10 ± 0.01a	8.04 ± 0.04a
12.5% quinoa biscuit	8.10 ± 0.10a	8.00 ± 0.01a	7.80 ± 0.15a	8.00 ± 0.00a	7.90 ± 0.10b	7.94 ± 0.04b
25% quinoa biscuit	7.70 ± 0.15b	7.50 ± 0.13ba	7.60 ± 0.16b	7.90 ± 0.23b	7.90 ± 0.18b	7.28 ± 0.11b

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

TABLE 8: Effect of the consumption of the 12.5% quinoa biscuit and 25% quinoa biscuit on body weight gain, weight gain%, food intake, and FER compared to control biscuit in diabetic rat groups.

Groups	Body weight gain (g)	Body weight gain (%)	Food intake (g)	FER
Positive control	25.71 ± 1.96c	21.42 ± 1.13c	17.40 ± 1.33a	0.0246 ± 0.002c
Control biscuit	23.55 ± 2.11cd	19.30 ± 0.77cd	17.33 ± 1.05a	0.0226 ± 0.001cd
12.5% quinoa biscuit	51.78 ± 4.66b	42.09 ± 3.21b	17.55 ± 1.21a	0.0491 ± 0.004b
25% quinoa biscuit	65.41 ± 5.40a	53.17 ± 4.03a	18.99 ± 1.10a	0.0574 ± 0.003a

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

addition of 50% or 100% quinoa flour addition biscuit formulations did not significantly change the biscuit properties compared to those made with wheat flour [43].

The results in Table 8 showed nonsignificant differences in body weight gain, body weight gain percent, and feed efficiency ratio (FER) between the diabetic positive rat group and the diabetic rat group that consumed control biscuit. On the other hand, the diabetic rat groups that consumed 12.5% quinoa biscuit and 25% quinoa biscuit showed a significant increase in body weight gain, body weight gain percent, and FER compared with the positive rat group. Among the experimental rat groups, the highest improvement in nutritional value appeared in the diabetic rat group that consumed 25% quinoa biscuits because of elevated lipid, protein, fiber, polyphenol, and antioxidant values compared to the control biscuit. The digestibility and absorption of nutrients were improved by quinoa fiber intake [44, 45]. Quinoa flour (QF) has been labelled a superfood because it contains more protein and has a better balanced amino acid composition than grain flours. QF is strong in protein, carbohydrate, fat, and fiber. Quinoa fiber is resistant to

enzymatic digestion and absorption in the small intestine but generally ferments entirely or partially in the large intestine. Dietary fiber is regarded to be necessary for excellent digestive health as well as a number of physiological benefits. Dietary fiber has been shown to reduce cholesterol and fat absorption, change postprandial insulin response, increase endogenous cholesterol conversion to bile acids, improve intestinal microbiota, and reduce the incidence and severity of gastrointestinal infection and inflammation [41]. Several prior studies in vitro have demonstrated that quinoa protein digestibility is higher than wheat protein digestion, which adds to its high stomach accessibility and digestion, as well as its health benefits [46].

The consumption of the tested biscuits showed a nonsignificant difference in blood hemoglobin in diabetic rat groups. Rats that consumed in the control group showed nonsignificant differences in glycosylated hemoglobin, insulin, and glucose compared with the positive control diabetic rat group. Following consumption of 12.5% quinoa biscuits and 25% quinoa biscuits for 60 days, there was a decline in blood glycosylated hemoglobin and glucose and

TABLE 9: Effect of consumption of the 12.5% quinoa biscuit and 25% quinoa biscuit on hemoglobin, glycosylated hemoglobin, insulin, and glucose compared to control biscuit in diabetic rat groups.

Groups	Hemoglobin (g/dl)	Glycosylated hemoglobin (%)	Insulin (ng/d)	Glucose (mg/dl)
Positive control	12.66 ± 1.21a	8.07 ± 1.03a	1.44 ± 0.14cd	295.11 ± 8.65a
Control biscuit	12.77 ± 1.09a	7.96 ± 0.78ab	1.54 ± 0.15c	289.98 ± 9.70ab
12.5% quinoa biscuit	13.75 ± 1.22a	6.11 ± 0.69c	2.08 ± 0.30b	129.66 ± 7.33c
25% quinoa biscuit	13.70 ± 1.13a	5.05 ± 0.55d	2.97 ± 0.42a	105.71 ± 5.14d

Mean values in each column having the same letter (a and b) are nonsignificantly different at  $P > 0.05$ .

an elevation in insulin compared with the positive control diabetic rat group. Moreover, the 25% quinoa biscuit rat group showed noticeable improvement in blood diabetic indicators, as shown in Table 9. The addition of quinoa flour to biscuits could increase the availability of fiber, which reduces the levels of blood lipids and glucose and elevates insulin sensitivity [47]. The intake of slowly digestible starches in whole grain flours from legumes and pseudocereals could lower glycemic indexes. The incorporation of quinoa flour into biscuits is beneficial in reducing glucose and increasing insulin without disturbing the nutritional balance owing to high phenol, fiber, and antioxidant activity. Quinoa seeds have anti-inflammatory, antioxidant, and antitumor actions due to the presence of valuable phenolic compounds [48–50].

#### 4. Conclusions

Incorporation of quinoa flour is beneficial for biscuit production to improve the rheological, technological, and sensory properties of biscuits because of the higher quantity of dietary fiber in addition to high nutritional values (protein, fiber, and other bioactive antioxidant compounds) and therapeutic value due to the hypoglycemic effect.

**4.1. Recommendations.** Regarding the results of our study, it is recommended to fortify bakery products, especially biscuits with quinoa flour, and advise their consumption by diabetic patients. Further studies are needed to produce products fortified with quinoa and sugar alternatives for diabetic patients.

#### Data Availability

The data used to support the findings of this study are available from the corresponding author upon request.

#### Additional Points

**Highlights.** (i) Quinoa is a new crop species with high nutritional value, so it acts as a complete food. (ii) Quinoa is incorporated into wheat flour at a definite percentage for biscuit production. (iii) It has been of interest to increase the nutritional and sensory quality of biscuits by incorporating functional foods to decline blood glycosylated hemoglobin and glucose and for an elevation in insulin level in the diabetic rat group.

#### Conflicts of Interest

The authors declare no conflicts of interest.

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## Research Article

# Physicochemical and Thermal Characteristics of Onion Skin from Fifteen Indian Cultivars for Possible Food Applications

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Every year tons of onion waste is produced worldwide. The dried outer onion skin contributed up to 70% of this waste. Outer-dried skins of fifteen prominent onion cultivars from India were selected for the study. A comparative study was done for proximate profiling, thermal characteristics, functional grouping, and mineral contents. Skin of cv. “NHRDF Red” contained the highest amount of crude protein ( $5.97 \pm 0.15$  g/100 g), ash ( $12.24 \pm 0.59$  g/100 g), and fiber ( $8.28 \pm 0.20$  g/100 g), whereas cv. “Pusa Red” possessed the highest amount of total fat ( $0.47 \pm 0.02$  g/100 g) and the maximum carbohydrates ( $76.66 \pm 0.56$  g/100 g) were found in “Pusa Riddhi.” Mineral analysis showed that cv. “NHRDF Red” had the maximum concentration of all 9 minerals along with sulphur content. Fourier transform infrared spectroscopy analysis explored the various metabolites present in each cultivar. The thermal analysis explored cv. “Agrifound Dark Red” as highly thermally stable having 70.98% residual mass. The lowest Tg temperature range was found between 64.4°C and 90.6°C for “Agrifound Dark Red.” Skin of cv. “NHRDF Red” was reported as the best source of protein, fiber, and minerals, which may be utilized for developing a food product.

## 1. Introduction

Onion (*Allium cepa* L.) is the second most important cultivated horticultural commodity after tomato. Globally, 99,968,016 tons of onion is produced from an area of 5,192,651 hectares [1]. China holds the first position in production with 18,122,435 tons and India is the second producer of onion (11,936,706 tons) in the world [1]. Up to 25% of qualitative and quantitative postharvest losses occurred during storage. Processing of onion has also increased which resulted in huge processing waste. Skin contributes maximum to processing waste. The European Union onion processing industry generates more than 500,000 tons of onion waste annually mainly in the UK, The

Netherlands, and Spain. This has become an environmental problem. The maximum amount of onion skin is generated by industries during peeling. This wasted skin is not suitable as fodder in higher concentrations due to its peculiar aroma [2].

Various studies explored onion skin as a rich source of polyphenols, antioxidants, fructooligosaccharides, and dietary fibers [3–5]. Moreover, onion skin extract has also been reported as an anticarcinogenic, hypocholesterolemic, good cardiovascular agent, and having an antiasthmatic effect [4, 5]. Onion flavonoids also show antiproliferative activity that can degrade excess polyamine concentration in the body [6]. Hence, onion skin powder may be incorporated into bakery products and bread which are good sources of energy



and consumed widely as well [7]. Wheat bread was fortified with the addition of 3% onion skin and it enhanced the antioxidant property of the bread with sensory acceptance [8]. Onion skin can also be utilized for the production of energy. Agricultural waste is the prime source to generate energy and is used as an important fuel after oil, coal, and natural gas worldwide [9]. The scarcity of oil, gas, and coal and the large availability of biomass provide an alternative to the industries for energy generation from biomass. The proper understanding of biomass characterization is essential for its effective utilization [10]. The proximate composition is an important parameter for developing a functional food or nutraceutical product. In addition to this, the thermal properties of onion skins are equally important to produce energy or biogas.

Overall, onion waste has become a disposable problem for onion processing industries because of its huge quantity that consequently creates environmental pollution. However, onion waste has the potential to be used as an active ingredient of a food or nutraceutical product. In this regard, the onion skin from 15 Indian cultivars was studied for the following objectives: (1) determining physicochemical properties, (2) analyzing mineral content and active functional groups, and (3) investigating thermal characteristics. All the parameters were studied to provide a solution to environmental pollution by exploring possible food applications.

## 2. Materials and Methods

**2.1. Plant Material and Samples Preparation.** Fifteen popular cultivars of onion ("Agrifound Dark Red," "Agrifound Light Red," "Arka Kirthiman," "Bhima Kiran," "Bhima Shakti," "Bhima Shubhra," "Hissar-2," "Hissar-3," "NHRDF Red," "Phursungi Local," "Pusa Madhavi," "Pusa Red," "Pusa Riddhi," "Sukhsagar," and "Udaipur Local") were procured from All India Network Research Project on Onion and Garlic, Division of Vegetable Sciences, Indian Agricultural Research Institute, New Delhi, India. The skin of the cultivars was collected (100 g each) after curing at an open field (20 days). Skins were washed with chlorinated water (0.5%) for the complete removal of dust and other impurities. Skins were washed again with distilled water, left open in a porous tray for 10 min to remove excess water, and then kept in deep freeze (Vestfrost Solutions, Denmark) at  $-40^{\circ}\text{C}$  for 24 h. Samples were freeze-dried using a lyophilizer (Mini Lyodel, Delvac Pumps, Chennai, India) with keeping the plate temperature at  $-50^{\circ}\text{C}$  with the pressure of 0.039 mbar and the drying process was continued up to 48 h. Freeze-dried skin was grounded using a mixer-grinder (3053 colts, Usha International Ltd., India) for powder formation and stored at  $-30^{\circ}\text{C}$  in airtight plastic containers for further use.

**2.2. Physicochemical Analysis.** Water activity ( $a_w$ ) of skin powder was analyzed by a water activity meter (Aqua LabTM-Dewpoint, US). The moisture content of the powder was analyzed by a moisture analyzer (Citizen®, India). Crude protein and total fat were estimated by Kjeldahl's

method (protein-nitrogen conversion factor 6.25) and the Soxhlet apparatus, respectively [10]. Ash content was obtained by placing 3 g sample in a muffle furnace at  $550^{\circ}\text{C}$  for 3 h; after cooling down, the sample was transferred in a dryer and calculated [11]. Total fiber concentration was estimated by calculating the difference in weights before calcination and after calcination. Each sample was digested with the solutions of sulphuric acid (1.25%) and sodium hydroxide (1.25%) and then the residue was calcined. Carbohydrate content was calculated by the following formula:

$$\begin{aligned} \text{Carbohydrate \%} = & 100 - \text{moisture content} + \text{total fiber} \\ & + \text{crude protein} + \text{total fat} + \text{ash content.} \end{aligned} \quad (1)$$

The total sulphur content was also analyzed by a CHNS analyzer (EURO Elemental Analyzer, Wegberg, Germany). The investigation was subjected to the total oxidation of the sample and complete combustion which changes the sample into  $\text{SO}_2$  (combustion product). The analysis was carried out according to Benitez et al. [12] with slight modifications. 1 mg sample of onion skin of each cultivar was taken for the analysis and sulfanilamide was used as a reference standard.

**2.3. Analysis of Mineral Content.** The onion skin powder was taken (0.5 g) into digestion tubes from each sample. Briefly, 2 mL of concentrated nitric acid ( $\text{HNO}_3$ ) was poured into each tube and placed into the digestion block at  $150^{\circ}\text{C}$ . After digestion, clear solutions were obtained which were used for mineral detection. Mineral concentration was detected by ICP-AES (inductive coupled plasma-atomic emission spectroscopy, Fujitsu Quality Lab Ltd., Japan). Results were expressed in mg/kg using external standards of K, Ca, P, Mn, Na, Mg, Fe, Zn, and Cu [13]. The calculation of adequate intake (AI) and population reference index (PRI) was done using the latest recommendations of the European Food Safety Authority (EFSA) [14]. Additionally, the recommended dietary allowances (RDA) given by the Food Safety and Standards Authority of India (FSSAI) for the minerals were also compared with the results [15].

**2.4. Assessment of Functional Groups.** The presence of various functional groups was assessed by FTIR (Fourier transform infrared spectroscopy, Agilent Cary 630, US) in transmission mode from  $4000$  to  $600\text{ cm}^{-1}$  as described by Kumar et al. [16]. The graphs were evaluated by MicroLab FTIR software.

**2.5. Thermal Characterization.** Mass loss of samples was estimated using TGA Libra (Netzsch, Germany) under nitrogen atmosphere (60 mL/min) at the heating rate of  $5^{\circ}\text{C}/\text{min}$  in the temperature range from  $20^{\circ}\text{C}$  to  $300^{\circ}\text{C}$  [17]. The sample mass used for each sample was  $4.0 \pm 0.1$  mg. The curves were analyzed by Netzsch Proteus software (version 6.1.10). Phase change with glass transition temperature was obtained by DSC 200 F3Maia (Netzsch, Germany). Sample ( $4.0 \pm 0.1$  mg) mass was packed in a hermetically sealed

aluminum pan in a nitrogen atmosphere (60 mL/min) at the heating rate of 5°C/min. The temperature range was taken from 30°C to 300°C and data analysis was carried out using Netzsch Proteus software (version 6.1.10).

**2.6. Statistical Analysis.** One-way analysis of variance (ANOVA) was applied for data analysis using IBM® SPSS statistics (version 20). All the samples were carried out in triplicate and the results were presented as mean  $\pm$  standard deviation on a dry weight basis. Moreover, the Principal Component Analysis (PCA) was applied to the results of proximate composition and mineral analysis to explore a comprehensive view of data.

### 3. Results and Discussion

**3.1. Physicochemical Analysis.** Water activity is an essential parameter, which is related to food safety, quality, and shelf life. Skin powder of cv. "Pusa Madhavi" had the highest water activity ( $0.40 \pm 0.01$ ), while "Agrifound Dark Red," "Arka Kirthiman," "Bhima Shakti," and "Pusa Red" cultivars exhibited the least water activity ( $0.09 \pm 0.01$ ). This indicates that cv. "Pusa Madhavi" contained excessive water for biological reactions in comparison to other cultivars, while cultivars with the least water activity had the lowest quantity of excess water. There was a significant difference ( $p \leq 0.05$ ) among cv. "Agrifound Dark Red," "Agrifound Light Red," "Bhima Shubhra," "Hissar-2," "Phursungi Local," "Pusa Madhavi," and "Udaipur Local" but no difference was observed among cv. "Bhima Kiran," "NHRDF Red," "Pusa Riddhi," and "Sukhsagar" (Table 1). Maximum moisture content was found in cv. "Pusa Madhavi" ( $13.23 \pm 0.90\%$  d.b.), while cv. "Pusa Riddhi" possessed the lowest moisture content with  $6.27 \pm 0.56\%$  d.b. Moisture content showed a significant difference ( $p \leq 0.05$ ) among "Agrifound Light Red," "Bhima Kiran," "Phursungi Local," "Pusa Red," "Pusa Madhavi," and "Pusa Riddhi," whereas cv. "Agrifound Dark Red," "Bhima Shakti," "Hissar-3," "NHRDF Red," "Phursungi Local," and "Sikh Sagar" were found to be at par with each other (Table 1). Compared to the onion bulb slices, Rapusas and Driscoll [18] reported 15.7% moisture content (wet basis) at  $2.23 \pm 0.09$  J/kg specific heat. In addition to this, Ismail et al. [19] analyzed Egyptian onion skins and found 3% moisture content on a dry basis. In addition to this, Pereira et al. [20] obtained  $9.41 \pm 0.09\%$  moisture content in dried onion waste. The differences in the fiber content of skin and cultivar types were the main reasons behind the variation in the moisture content. In addition, moisture content also depends upon the water available in the sample and the vapor pressure in the atmosphere [21].

Various studies revealed the proximate composition of onion bulbs [18, 19] but there are very few studies available that explore the proximate components of onion skins. Present research for proximate analysis showed that skin of "NHRDF Red" cultivar contained the highest amount of crude protein with  $5.97 \pm 0.15$  g/100 g and the lowest concentration was reported in "Udaipur Local" ( $3.90 \pm 0.10$  g/

100 g). "Pusa Red" exhibited the highest content of total fat ( $0.47 \pm 0.02$  g/100 g) among all the cultivars, while the least concentration was found in "Bhima Shubhra" ( $0.30 \pm 0.02$  g/100 g). The harvesting conditions and the types of cultivars may impart the difference in protein content. A significant difference ( $p \leq 0.05$ ) was found among "Bhima Shubhra," "Hissar-3," "Pusa Red," and "Pusa Riddhi" cultivars, whereas "Agrifound Dark Red," "Agrifound Light Red," "Arka Kirthiman," "Bhima Kiran," "Pusa Madhavi," "Pusa Riddhi," and "Sukhsagar" were found to be at par with each other for total fat content (Table 1).

The highest amount of crude fiber was obtained in "NHRDF Red" ( $8.28 \pm 0.20$  g/100 g), while the lowest crude fiber content ( $4.45 \pm 0.44$  g/100 g) was recorded in "Pusa Madhavi." There was a significant difference ( $p \leq 0.05$ ) among "Agrifound Dark Red," "Agrifound Light Red," "Bhima Kiran," "NHRDF Red," and "Pusa Madhavi" cultivars for crude fiber content. The highest ash content ( $12.24 \pm 0.59$  g/100 g) was recorded in cv. "NHRDF Red" skin and the lowest concentration of ash was reported in cv. "Pusa Riddhi" ( $6.88 \pm 0.39$  g/100 g). Near approximate concentration ( $5.7 \pm 0.3$  g/100 g) of ash was reported in dry weight of onion skin waste [22]. "Agrifound Dark Red," "Hissar-3," "Phursungi Local," and "Sukhsagar" were found at par with regard to ash content and a significant difference was found among "Bhima Kiran," "Bhima Shubhra," "Phursungi Local," "Pusa Madhavi," "Pusa Riddhi," and "Pusa Red." The concentration of biomolecules may vary with the cultivar types, soil composition, and harvesting time. Maximum carbohydrate content ( $76.66 \pm 0.56$  g/100 g) was obtained in cv. "Pusa Riddhi," which was much higher than onion slices ( $14.77 \pm 0.04$  g/100 g) and the extract of onion bulb ( $6.91 \pm 0.02$  g/100 g) [18]. The lowest amount of carbohydrate was recorded in "NHRDF Red" cultivar ( $64.32 \pm 0.99$  g/100 g). A significant difference ( $p \leq 0.05$ ) was found among cv. "Agrifound Dark Red," "Agrifound Light Red," "Bhima Shakti," "Bhima Shubhra," "NHRDF Red," "Pusa Madhavi," and "Udaipur Local" (Table 1). Majid et al. [23] have analyzed onion bulb powder of sprouted and unsprouted cultivars and obtained 3.087–6.08% total fibers, 70.74–79.54% carbohydrate, and 8.57–14.16% proteins. The proximate analysis showed that the color of skin, that is, whited, light red, red, and dark red, imparted the variations in the proximate composition of cultivars. This study showed that onion skin and bulb powder are comparable for total fibers, total carbohydrates, and proteins. Also, the skin is as important for fiber, carbohydrates, and proteins as bulbs.

The highest sulphur concentration was obtained in the skin of cv. "NHRDF Red" ( $8.63 \pm 0.17\%$ ) and lowest in cv. "Agrifound Light Red" ( $3.48 \pm 0.24\%$ ), while sulphur was not detected in cv. "Bhima Shubhra" and "Udaipur Local." All the cultivars were significantly different ( $p \leq 0.05$ ) from each other except "Arka Kirthiman" and "Sukhsagar" which were at par. Previous studies revealed that the outer skin of onion contains the lowest amount ( $15.6 \pm 0.6$   $\mu$ moles/g dw) of sulphur compared to its other sections such as inner scale ( $153.1 \pm 5$   $\mu$ moles/g dw), top-bottom ( $143.8 \pm 3.3$   $\mu$ moles/g dw), and onion bulb ( $121.9 \pm 3.2$   $\mu$ moles/g dw) [12].

TABLE 1: Physicochemical composition of onion skin powder of fifteen cultivars.

Cultivar	Water activity ( $a_w$ )	Moisture content (%)	Crude protein (%)	Total fat (%)	Crude fiber (%)	Ash content (%)	Carbohydrate (%)	Sulphur content (%)
Agrifound Dark Red	0.09 ± 0.00 <sup>a</sup>	8.77 ± 0.49 <sup>bc</sup>	5.73 ± 0.31 <sup>gh</sup>	0.35 ± 0.05 <sup>ab</sup>	6.53 ± 0.42 <sup>ef</sup>	9.53 ± 0.35 <sup>cd</sup>	69.08 ± 1.16 <sup>cd</sup>	4.50 ± 0.17 <sup>g</sup>
Agrifound Light Red	0.35 ± 0.01 <sup>g</sup>	11.24 ± 1.07 <sup>e</sup>	5.83 ± 0.15 <sup>gh</sup>	0.32 ± 0.02 <sup>ab</sup>	5.35 ± 0.13 <sup>bc</sup>	7.89 ± 0.51 <sup>b</sup>	69.37 ± 1.02 <sup>de</sup>	3.48 ± 0.24 <sup>d</sup>
Arka Kirthiman	0.09 ± 0.01 <sup>a</sup>	10.88 ± 0.35 <sup>de</sup>	5.27 ± 0.31 <sup>e</sup>	0.35 ± 0.03 <sup>ab</sup>	6.96 ± 0.62 <sup>f</sup>	9.11 ± 0.14 <sup>c</sup>	67.44 ± 0.81 <sup>b</sup>	4.09 ± 0.03 <sup>f</sup>
Bhima Kiran	0.13 ± 0.01 <sup>ab</sup>	7.89 ± 0.31 <sup>b</sup>	4.27 ± 0.15 <sup>bc</sup>	0.34 ± 0.04 <sup>ab</sup>	5.67 ± 0.32 <sup>cd</sup>	11.60 ± 0.46 <sup>gh</sup>	70.20 ± 0.57 <sup>de</sup>	2.87 ± 0.08 <sup>c</sup>
Bhima Shakti	0.09 ± 0.00 <sup>a</sup>	8.79 ± 0.44 <sup>bc</sup>	4.37 ± 0.15 <sup>bc</sup>	0.41 ± 0.02 <sup>cd</sup>	5.05 ± 0.23 <sup>abc</sup>	6.90 ± 0.20 <sup>a</sup>	74.48 ± 0.79 <sup>f</sup>	5.08 ± 0.08 <sup>h</sup>
Bhima Shubhra	0.14 ± 0.01 <sup>bc</sup>	6.46 ± 0.47 <sup>a</sup>	3.93 ± 0.15 <sup>a</sup>	0.30 ± 0.02 <sup>a</sup>	5.49 ± 0.49 <sup>bcd</sup>	7.83 ± 0.25 <sup>b</sup>	75.89 ± 0.20 <sup>g</sup>	n.d.
Hissar-2	0.30 ± 0.01 <sup>f</sup>	10.76 ± 0.67 <sup>de</sup>	5.40 ± 0.20 <sup>ef</sup>	0.45 ± 0.05 <sup>de</sup>	5.07 ± 0.15 <sup>abc</sup>	10.93 ± 0.15 <sup>fg</sup>	67.39 ± 0.79 <sup>b</sup>	6.72 ± 0.17 <sup>l</sup>
Hissar-3	0.21 ± 0.02 <sup>de</sup>	8.88 ± 0.29 <sup>bc</sup>	4.43 ± 0.21 <sup>c</sup>	0.41 ± 0.02 <sup>cd</sup>	7.03 ± 0.43 <sup>f</sup>	10.07 ± 0.15 <sup>d</sup>	69.81 ± 0.34 <sup>cd</sup>	5.68 ± 0.21 <sup>j</sup>
NHRDF Red	0.17 ± 0.05 <sup>bc</sup>	8.76 ± 1.24 <sup>bc</sup>	5.97 ± 0.15 <sup>h</sup>	0.43 ± 0.03 <sup>de</sup>	8.28 ± 0.20 <sup>g</sup>	12.24 ± 0.59 <sup>h</sup>	64.32 ± 0.99 <sup>a</sup>	8.63 ± 0.17 <sup>m</sup>
Phursungi Local	0.24 ± 0.04 <sup>e</sup>	9.87 ± 0.50 <sup>cd</sup>	4.30 ± 0.10 <sup>bc</sup>	0.43 ± 0.02 <sup>de</sup>	4.80 ± 0.26 <sup>ab</sup>	9.84 ± 0.31 <sup>d</sup>	70.77 ± 0.89 <sup>e</sup>	5.30 ± 0.17 <sup>i</sup>
Pusa Madhavi	0.40 ± 0.01 <sup>h</sup>	13.23 ± 0.90 <sup>f</sup>	4.10 ± 0.10 <sup>abc</sup>	0.36 ± 0.02 <sup>b</sup>	4.45 ± 0.44 <sup>a</sup>	10.85 ± 0.71 <sup>ef</sup>	67.02 ± 0.73 <sup>b</sup>	6.11 ± 0.04 <sup>k</sup>
Pusa red	0.09 ± 0.02 <sup>a</sup>	10.33 ± 0.95 <sup>de</sup>	4.03 ± 0.15 <sup>ab</sup>	0.47 ± 0.02 <sup>e</sup>	5.67 ± 0.42 <sup>cd</sup>	8.88 ± 0.69 <sup>c</sup>	70.62 ± 1.63 <sup>de</sup>	2.61 ± 0.14 <sup>b</sup>
Pusa Riddhi	0.16 ± 0.04 <sup>bc</sup>	6.27 ± 0.56 <sup>a</sup>	4.80 ± 0.10 <sup>d</sup>	0.36 ± 0.02 <sup>bc</sup>	5.04 ± 0.81 <sup>abc</sup>	6.88 ± 0.39 <sup>a</sup>	76.66 ± 0.56 <sup>g</sup>	3.82 ± 0.06 <sup>e</sup>
Sukhsagar	0.13 ± 0.02 <sup>b</sup>	9.98 ± 0.11 <sup>cd</sup>	5.60 ± 0.20 <sup>fg</sup>	0.33 ± 0.03 <sup>ab</sup>	6.17 ± 0.31 <sup>de</sup>	10.18 ± 0.31 <sup>de</sup>	67.74 ± 0.53 <sup>bc</sup>	4.15 ± 0.04 <sup>f</sup>
Udaipur Local	0.18 ± 0.01 <sup>cd</sup>	6.76 ± 0.43 <sup>a</sup>	3.90 ± 0.10 <sup>a</sup>	0.31 ± 0.02 <sup>a</sup>	4.57 ± 0.35 <sup>a</sup>	8.03 ± 0.15 <sup>b</sup>	76.43 ± 0.69 <sup>g</sup>	n.d.

Values are mean ± standard deviation ( $n=3$ ). Values with different superscripts <sup>ah</sup> in the same column are significantly different ( $p \leq 0.05$ ) by Duncan's multiple range test.

However, sulphur is an integrated part of ACSOs (S-alk(en)yl-L-cysteine sulfoxides), which are onion flavor precursors, but in various studies, no correlation was reported between the concentration of flavor precursors and total sulphur content, which revealed that there was a poor correlation between pungency and sulphur accumulation. The percentage of S-ACSOs in total sulphur content showed an increasing pattern from the outer section to the inner section of the skin; however, brown outer skin contained almost 30% of the total sulphur content in flavor precursors [12]. Benitez et al. [12] found the lowest flavor precursors in brown skin compared to onion bulb. On the other hand, Sarkar et al. [24] reported 0.44 ± 0.01% of sulphur concentration in onion bulb produced by different mulching practices. It is already proven that mulching and other preharvest factors may affect sulphur content in onion bulb [25]. Concentration and ratio of flavor precursors differ from cultivar to cultivar and its ratio imparts peculiar aroma and taste to the onion.

**3.2. Mineral Analysis.** The mineral composition of onion skin powder of fifteen cultivars was determined on a dry weight basis. K, P, and Ca were recorded as principal abundant macrominerals in the outermost peel, ranging from 877.82 ± 38.79 to 3474.01 ± 17.16 mg/kg, 796.22 ± 13.01 to 2873.10 ± 46.76 mg/kg, and 762.42 ± 38.21 to 3007.81 ± 16.42 mg/kg, respectively. The study revealed that cv. "NHRDF Red" contained the highest amount of macrominerals, such as K (3474.01 ± 17.16 mg/kg), Ca (3007.81 ± 16.42 mg/kg), P (2873.10 ± 46.76 mg/kg), and Mg (1034.54 ± 49.98 mg/kg) among all the cultivars. The least amount of macronutrients was obtained in cv. "Bhima Shubhra" and "Udaipur Local" (Table 2). However, all cultivars were found to be a valuable source of important minerals according to the RDA of EFSA and FSSAI [14, 15].

According to FSSAI, RDA for women-men should be 3225–3750, 600, 600, and 310–340 mg/d for K, P, Ca, and Mg, respectively (Table 2). The variation in mineral content of cultivars might be due to soil composition, harvesting time, and varietal differences.

Among the trace elements, Fe, Zn, and Cu were found in the highest concentration. They were quantified in the range of 110.60 ± 0.37–211.96 ± 0.08 mg/kg, 17.95 ± 0.06–39.27 ± 0.34 mg/kg, and 12.45 ± 0.43–29.49 ± 0.32 mg/kg, respectively. Cultivar "NHRDF Red" was reported to have the highest concentration of trace elements among all the cultivars. The lowest concentration of trace elements was obtained in cv. "Bhima Shubhra." Cultivars "Agrifound Dark Red," "Bhima Shakti," "Hissar-2," "Hissar-3," "NHRDF Red," "Pusa Madhavi," and "Pusa Red" were found significantly different ( $P \leq 0.05$ ) from each other, whereas no significant ( $p \leq 0.05$ ) difference was obtained between cultivars "Agrifound Light Red," "Arka Kirthiman," "Bhima Kiran," "Bhima Shubhra," "Phursungi Local," and "Sukhsagar." A previous study showed a similar amount of macrominerals such as K (7297.88 mg/kg), P (2491.04 mg/kg), Ca (1824.29 mg/kg), and Mg (1990.59 mg/kg) and a lower range for trace elements, that is, Fe (160.69 mg/kg), Al (41.46 mg/kg), Zn (38.44 mg/kg), and Cu (27.23 mg/kg) in onion bulb [26]. Choje and Terry [27] analyzed different cultivars of onion and obtained a greater range of macrominerals, that is, K (149.00–270.85 mg/100 g), P (15.92–29.25 mg/100 g), Na (3.84–14.57 mg/100 g), and Mg (6.39–10.81 mg/100 g), while Mn (0.11–0.23 mg/100 g) and Zn (0.27–0.45 mg/100 g) were recorded in a lower concentration. Bello et al. [28] analyzed onion bulbs and found a good concentration of K (2.98 mg/100 g), Ca (1.22 mg/100 g), Mn (0.05 mg/100 g), Fe (0.04 mg/100 g), and Cu (0.13 mg/100 g) but lower than skin because onion skin contains a concentrated amount of minerals compared to bulb due to the absence of water. Likewise, Sarkar et al. [24]

TABLE 2: Mineral composition of onion skins of fifteen cultivars.

Cultivar	K	Ca	P	Mn	Na	Mg	Fe	Zn	Cu
Agrifound Dark Red	3366.17 ± 28.29 <sup>k</sup>	1197.18 ± 7.82 <sup>b</sup>	2418.49 ± 66.34 <sup>ef</sup>	11.67 ± 0.41 <sup>d</sup>	508.34 ± 17.46 <sup>de</sup>	762.18 ± 25.33 <sup>h</sup>	170.87 ± 0.29 <sup>j</sup>	34.54 ± 0.39 <sup>i</sup>	25.52 ± 0.39 <sup>i</sup>
Agrifound Light Red	3050.16 ± 42.48 <sup>h</sup>	1381.58 ± 23.57 <sup>d</sup>	2551.78 ± 41.45 <sup>f</sup>	12.55 ± 0.51 <sup>e</sup>	610.89 ± 20.85 <sup>g</sup>	335.27 ± 9.42 <sup>c</sup>	150.90 ± 0.13 <sup>g</sup>	29.65 ± 0.28 <sup>g</sup>	21.78 ± 0.29 <sup>f</sup>
Arka Kirthiman	2137.99 ± 56.22 <sup>c</sup>	1222.92 ± 29.35 <sup>b</sup>	2056.44 ± 43.67 <sup>b</sup>	11.67 ± 0.41 <sup>cd</sup>	575.46 ± 42.74 <sup>g</sup>	207.63 ± 6.06 <sup>a</sup>	135.86 ± 0.17 <sup>d</sup>	21.71 ± 0.23 <sup>c</sup>	16.52 ± 0.34 <sup>c</sup>
Bhima Kiran	2614.45 ± 78.93 <sup>c</sup>	1325.89 ± 28.02 <sup>c</sup>	2632.97 ± 55.48 <sup>g</sup>	13.44 ± 0.45 <sup>fg</sup>	674.82 ± 28.11 <sup>h</sup>	297.96 ± 7.29 <sup>bc</sup>	130.74 ± 0.17 <sup>c</sup>	22.62 ± 0.19 <sup>d</sup>	18.66 ± 0.12 <sup>d</sup>
Bhima Shakti	3140.76 ± 56.26 <sup>i</sup>	1492.81 ± 18.16 <sup>e</sup>	2458.65 ± 52.66 <sup>f</sup>	14.03 ± 0.17 <sup>gh</sup>	488.47 ± 20.31 <sup>d</sup>	361.48 ± 32.46 <sup>de</sup>	140.82 ± 0.24 <sup>e</sup>	25.56 ± 0.29 <sup>c</sup>	23.49 ± 0.27 <sup>g</sup>
Bhima Shubhra	959.05 ± 27.72 <sup>b</sup>	770.48 ± 30.61 <sup>a</sup>	796.22 ± 13.01 <sup>a</sup>	10.04 ± 0.17 <sup>b</sup>	127.97 ± 1.52 <sup>a</sup>	168.57 ± 11.23 <sup>a</sup>	110.60 ± 0.37 <sup>a</sup>	17.95 ± 0.06 <sup>a</sup>	12.45 ± 0.43 <sup>a</sup>
Hissar-2	3250.05 ± 36.78 <sup>j</sup>	2892.29 ± 23.28 <sup>j</sup>	2789.47 ± 20.34 <sup>h</sup>	17.37 ± 0.57 <sup>i</sup>	794.25 ± 10.17 <sup>i</sup>	888.78 ± 16.61 <sup>i</sup>	197.01 ± 0.11 <sup>n</sup>	38.39 ± 0.34 <sup>m</sup>	27.95 ± 0.14 <sup>k</sup>
Hissar-3	2667.44 ± 27.70 <sup>e</sup>	2496.18 ± 72.00 <sup>h</sup>	2430.87 ± 50.50 <sup>de</sup>	14.09 ± 0.17 <sup>gh</sup>	599.86 ± 10.8 <sup>g</sup>	641.32 ± 41.48 <sup>g</sup>	181.07 ± 0.84 <sup>i</sup>	31.48 ± 0.23 <sup>h</sup>	24.29 ± 0.25 <sup>h</sup>
NHRDF Red	3474.01 ± 17.16 <sup>j</sup>	3007.81 ± 16.42 <sup>k</sup>	2873.10 ± 46.76 <sup>i</sup>	18.68 ± 0.42 <sup>j</sup>	879.97 ± 26.15 <sup>j</sup>	1034.54 ± 49.98 <sup>j</sup>	211.96 ± 0.08 <sup>o</sup>	39.27 ± 0.34 <sup>n</sup>	29.49 ± 0.32 <sup>l</sup>
Phursungi Local	2233.70 ± 18.23 <sup>d</sup>	2691.22 ± 32.91 <sup>i</sup>	2395.82 ± 55.92 <sup>de</sup>	13.33 ± 0.48 <sup>fg</sup>	431.88 ± 32.34 <sup>c</sup>	443.80 ± 39.69 <sup>f</sup>	160.53 ± 0.33 <sup>h</sup>	33.61 ± 0.40 <sup>i</sup>	21.36 ± 0.29 <sup>f</sup>
Pusa Madhavi	2767.56 ± 30.22 <sup>f</sup>	2702.44 ± 26.78 <sup>i</sup>	2362.49 ± 17.24 <sup>cd</sup>	14.37 ± 0.47 <sup>h</sup>	540.69 ± 45.12 <sup>ef</sup>	743.15 ± 27.33 <sup>h</sup>	175.85 ± 0.18 <sup>k</sup>	35.41 ± 0.42 <sup>k</sup>	24.41 ± 0.20 <sup>h</sup>
Pusa Red	2855.08 ± 21.63 <sup>g</sup>	2699.48 ± 11.41 <sup>i</sup>	2466.60 ± 55.19 <sup>e</sup>	13.26 ± 0.42 <sup>f</sup>	496.23 ± 7.62 <sup>d</sup>	856.01 ± 26.17 <sup>i</sup>	181.72 ± 0.25 <sup>m</sup>	37.46 ± 0.43 <sup>l</sup>	26.10 ± 0.04 <sup>j</sup>
Pusa Riddhi	2662.61 ± 22.77 <sup>e</sup>	2381.53 ± 23.86 <sup>g</sup>	2300.07 ± 11.92 <sup>c</sup>	12.27 ± 0.34 <sup>e</sup>	404.02 ± 5.94 <sup>c</sup>	397.96 ± 8.69 <sup>e</sup>	161.35 ± 0.16 <sup>i</sup>	27.55 ± 0.39 <sup>f</sup>	21.42 ± 0.18 <sup>f</sup>
Sukhsagar	2188.48 ± 10.82 <sup>cd</sup>	2107.15 ± 23.17 <sup>f</sup>	2125.58 ± 81.74 <sup>b</sup>	11.20 ± 0.61 <sup>c</sup>	298.38 ± 8.39 <sup>b</sup>	281.13 ± 19.20 <sup>b</sup>	143.55 ± 0.32 <sup>f</sup>	21.39 ± 0.30 <sup>c</sup>	19.93 ± 0.19 <sup>e</sup>
Udaipur Local	877.82 ± 38.79 <sup>a</sup>	762.42 ± 38.21 <sup>a</sup>	870.27 ± 32.49 <sup>a</sup>	9.11 ± 0.41 <sup>a</sup>	121.07 ± 0.56 <sup>a</sup>	188.16 ± 11.85 <sup>a</sup>	125.80 ± 0.34 <sup>b</sup>	19.55 ± 0.29 <sup>b</sup>	13.42 ± 0.41 <sup>b</sup>
%AI/PRI*	124	12	—	126	—	80	191	60	114
RDA (mg/d)#	3225–3750	600	600	4	1900–2100	310–340	17–21	10–12	1.7

Data are mean value ± standard deviation (n = 3) in mg/kg. \* As per the recommendations of EFSA [14]. # As per the recommendations of FSSAI [15]. —: not available. Values in the same column with different letters are significantly different (P ≤ 0.05) by Duncan's multiple range test.

reported 1.10, 0.29, and 0.26 mg/100 g concentrations of K, P, and Ca in onion bulb cultivated by different mulching techniques, respectively. It showed that intrinsic and extrinsic factors influence the level of minerals. In Japan, onion bulbs from five major growing areas were investigated and a higher range of Na ( $107 \pm 45$ – $343 \pm 221$   $\mu\text{g/g}$ ), P ( $2530 \pm 423$ – $3120 \pm 498$   $\mu\text{g/g}$ ), and Mg ( $798 \pm 131$ – $948 \pm 209$   $\mu\text{g/g}$ ) was reported. Moreover, Ariyama et al. [29] analyzed Japanese onions for Zn and quantified them in the range of  $12.8 \pm 3.0$ – $17.9 \pm 7.2$   $\mu\text{g/g}$ . Geographical locations, cultivar types, and growing conditions play an important role in the mineral composition of the onion bulb and skin [26, 27].

**3.3. Principal Component Analysis.** PCA was performed to identify and visualize the association of the proximate composition and mineral composition in the fifteen onion cultivars that were considered for this study. The results of the PCA analysis of proximate composition showed that two PCs (Dim1 and Dim2) could explain 68.6% of the variability in data (Figure 1(a)). The fat, ash, moisture content, and crude protein showed a positive correlation, while carbohydrate showed a negative correlation with all the other parameters. Moreover, an inverse correlation was also observed between fiber content and water activity. This result was expected since fibers can bind the water molecules within their complex matrix and reduce the overall vapor pressure, thereby preventing it from being detected by the water activity meter. It can also be noted that Udaipur Local, Bhima Shakti, Pusa Riddhi, and Bhima Shubhra have considerably higher carbohydrate content, while Hissar-3, Sukhsagar, Arka Kirthiman, and Agrifound Dark Red had higher fiber and crude protein.

Based on the biplot of the mineral composition (Figure 1(b)), it can be stated that the first principal component (Dim1) explained 100% of the variability in mineral data. The sodium, potassium, and phosphorus content exhibited a negative correlation with the iron, potassium, copper, and zinc content in the cultivars. This shows that the former set of mineral parameters could be a good indicator of the latter set and vice versa. However, the variation in the manganese content for the cultivars did not show any correlation to the other two mineral correlation sets and showed less variability (Dim2 = 0%). It should also be noted that the onion varieties that are clustered together show a similar mineral composition. For instance, the varieties Phursungi Local, Udaipur Local, Pusa Riddhi, and Sukhsagar would show homogeneous properties. Similarly, Pusa Madhavi, Bhima Shakti, NHRDF Red, and Hissar-3 would have a similar mineral composition. Another cluster that can be recognized consists of the Pusa Red, Arka Kirthiman, Bhima Kiran, and Bhima Shubhra varieties; however, the correlation between these varieties would be relatively less as compared to the other two identified clusters due to a more scattered cluster.

**3.4. Functional Group Assessment.** FTIR spectroscopy is the best-suited technique to investigate the probable chemical compounds or metabolites in samples. It is a nondestructive

method of analysis, which saves the sample contents from mechanical or thermal energy [30]. In the present study, FTIR analysis of onion skin powder of fifteen cultivars confirmed the presence of various functional groups. Spectra of different cultivars are shown in Figure 2. Spectral bands between fingerprint regions from  $1800$  to  $750$   $\text{cm}^{-1}$  reflect the primary biomolecules like protein, carbohydrate, lipid, nucleic acid, and polyphenols [31]. The skin powder of all the cultivars showed a strong and common absorbance between the wavelength of  $1012$ – $1009$   $\text{cm}^{-1}$  and  $1607$ – $1597$   $\text{cm}^{-1}$ , while medium absorbance was obtained between the wave number of  $1418$ – $1317$   $\text{cm}^{-1}$ ,  $2116$ – $2113$   $\text{cm}^{-1}$ , and  $2917$ – $2900$   $\text{cm}^{-1}$ . The weak wavelength ranged from  $1147$  to  $1100$   $\text{cm}^{-1}$ ,  $1245$  to  $1237$   $\text{cm}^{-1}$ ,  $1878$  to  $1849$   $\text{cm}^{-1}$ , and  $2342$  to  $2320$   $\text{cm}^{-1}$  in all the cultivars. Apart from this, other absorbance ranges were also observed in some cultivars between  $840$  and  $804$   $\text{cm}^{-1}$  wavelengths. Both strong absorbances that fall between the wavelength of  $1150$ – $1000$   $\text{cm}^{-1}$  and  $1615$ – $1495$   $\text{cm}^{-1}$  are assigned to C-F stretch aliphatic organic halogen compounds and aromatic nitro compounds, respectively [32]. Medium peak frequencies fall between the range of  $1420$ – $1300$   $\text{cm}^{-1}$ ,  $2140$ – $2100$   $\text{cm}^{-1}$ , and  $2923$ – $2915$   $\text{cm}^{-1}$ , which showed the presence of carboxylate (carboxylic acid salt),  $\text{C}\equiv\text{C}$  stretch terminal alkyne, and methylene in the saturated aliphatic group, respectively [33]. Weak frequencies between  $1200$  and  $1100$   $\text{cm}^{-1}$ ,  $1850$  and  $1650$   $\text{cm}^{-1}$ , and  $2363$  and  $2313$   $\text{cm}^{-1}$ , respectively, represent sulfonates (sulphur oxy compounds), carbonyl compounds, and atmospheric  $\text{CO}_2$ . The other captured peaks between  $900$  and  $800$   $\text{cm}^{-1}$  are assigned to the C-H stretch of aromatic (aryl) ring frequency [31]. The study showed that all the cultivars exhibited a similar pattern of peaks; however, the peak sharpness (strong and medium) showed a difference in biomolecules level of onion due to varietal difference. Larrosa et al. [34] investigated vegetable paste and reported the presence of phenolic, aromatic, and carbonic groups. Characterization of red onion skin tannin represented almost the same peaks absorbance at  $1650$   $\text{cm}^{-1}$ ,  $1437$   $\text{cm}^{-1}$ , and  $1115$   $\text{cm}^{-1}$  [35]. Moreover, the FTIR study of onion powder showed the presence of starch and other important compounds between the wavenumbers of  $4000$ – $650$   $\text{cm}^{-1}$ , which supports the present study [36].

**3.5. Thermal Characterization.** The thermal decomposition analysis was carried out by TGA for the detection of mass change/loss of samples. TGA curves are shown in Figure 3. Thermograms of TGA showed that the highest mass loss (25.31%) was reported in cv. “Phursungi Local” (Figure 3). A total 1.91% residual mass was obtained for “Phursungi Local.” The least mass decomposition (0.64%) was observed in cv. “Agrifound Dark Red” between the temperature ranges of  $220^\circ\text{C}$ – $270^\circ\text{C}$  with the residual mass of 70.98%. The maximum mass loss and total residual mass of each cultivar are presented in Table 3. A significant ( $p \leq 0.05$ ) difference was obtained between cultivars. Mass loss at the first step was probably due to the evaporation of available water.

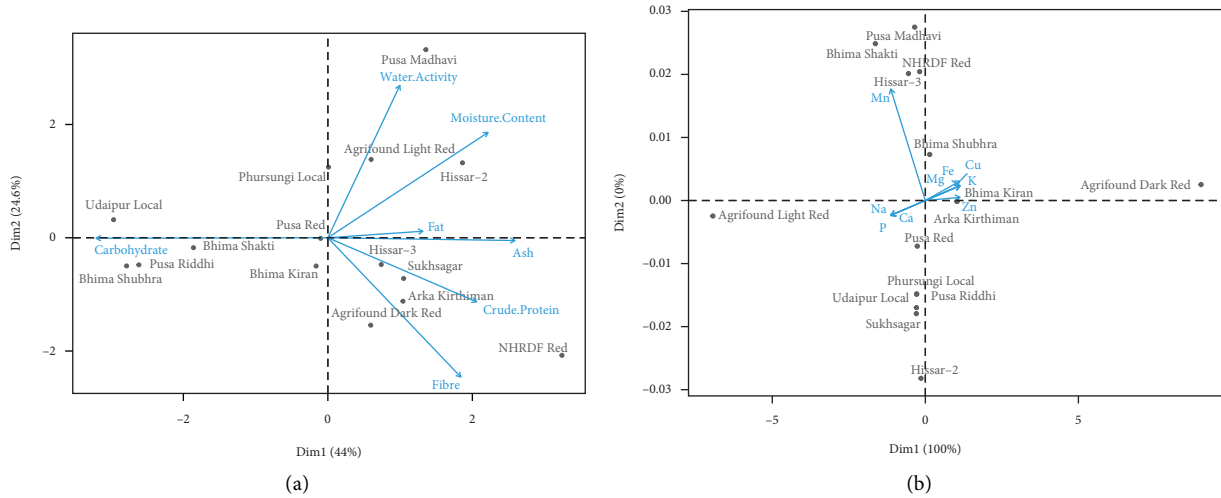


FIGURE 1: Principal Component Analysis biplots for (a) proximate composition and (b) mineral content.

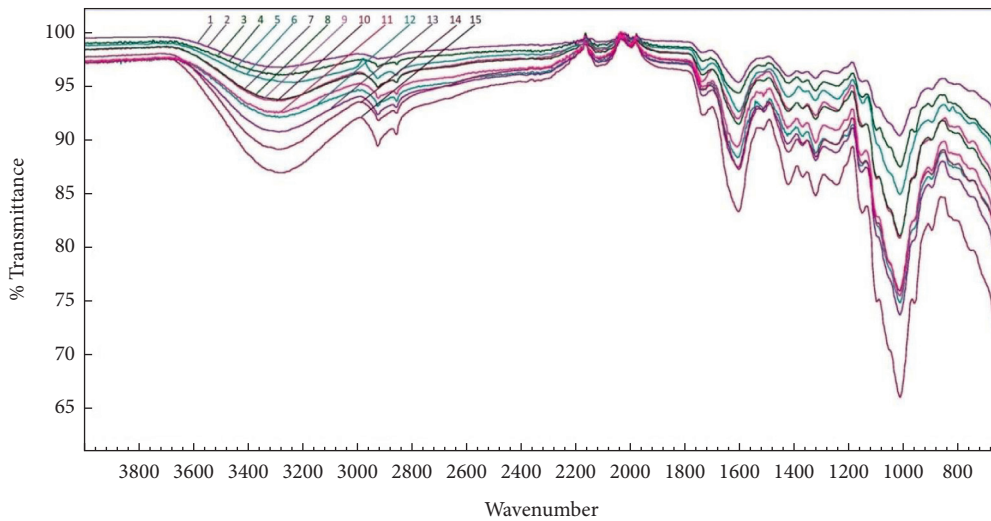


FIGURE 2: IR spectra of onion skin powder of fifteen cultivars: 1, Agrifound Dark Red; 2, Agrifound Light Red; 3, Arka Kirthiman; 4, Bhima Kiran; 5, Bhima Shakti; 6, Bhima Shubhra; 7, Hissar-2; 8, Hissar-3; 9, NHRDF Red; 10, Phursungi Local; 11, Pusa Madhavi; 12, Pusa Red; 13, Pusa Riddhi; 14, Sukhsagar; 15, Udaipur Local.

At the other decomposition steps, the mass loss occurred probably because of different components such as secondary metabolites and carbohydrates [37]. At the end of the experiment (299.9°C), the lowest residual mass with 1.91% was found in cv. “Phursungi Local,” while the highest residual mass was obtained in cv. “Agrifound Dark Red” with 70.98% Table 3. To the best of our knowledge, previous studies are not available on the thermal attributes of onion skin powder. However, a TGA study of the powder of guava, sapota, and papaya showed the maximum mass loss from 30% to 41.45% between combined temperature ranges of 132°C–332°C [36], which supports the findings of the present investigation. Moreover, the thermal analysis of the extract of *Syzygium cumini* L. leaves also revealed a significant mass loss (28.16%) between the temperature range of 209°C–260°C [38]. Different concentrations of crude fiber, ash, and protein are the main reasons for differences among residual

masses of different cultivars. TGA revealed “Phursungi Local” as the least thermally stable cultivar and “Agrifound Dark Red” as the best thermally stable cultivar which explores that the onion skin powder of cv. “Agrifound Dark Red” can be utilized for higher temperature processes.

DSC curves explored the phase change and glass transition temperature (T<sub>g</sub>) of onion skin powder of fifteen cultivars. DSC curves are given in Figure 4 and T<sub>g</sub> for all the cultivars is given in Table 4. The cv. “Agrifound Dark Red” had a T<sub>g</sub> temperature between 64.4°C and 90.6°C. It also showed a single endothermic peak at 102.1°C (Figure 4). “Agrifound Light Red” showed the T<sub>g</sub> between 135.9°C and 140.9°C. It also showed two endothermic peaks: the first huge at 160.4°C and the second small peak at 244°C. The T<sub>g</sub> temperature for cv. “Arka Kirthiman” was recorded between the temperature ranges of 111.1°C–132.7°C. The T<sub>g</sub> of cv. “NHRDF Red” was found between 120.2°C and 124.5°C and

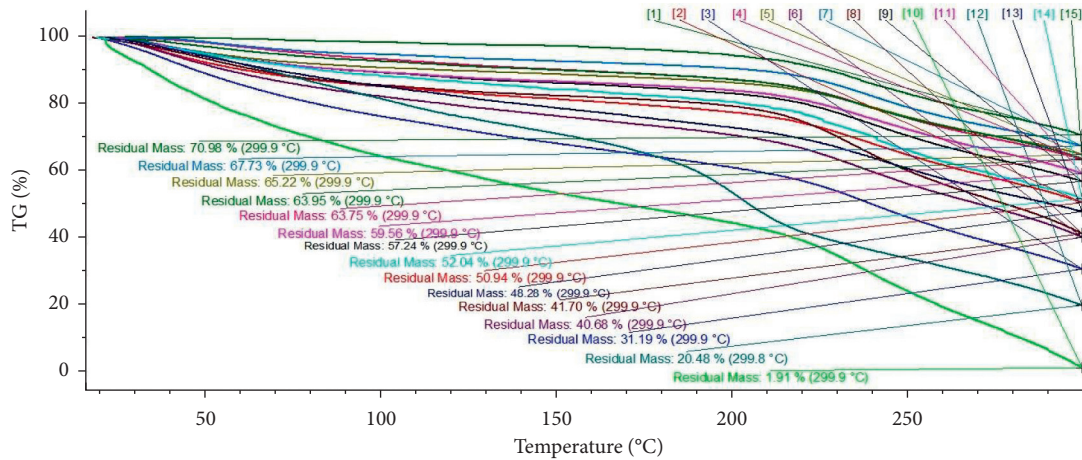


FIGURE 3: Combined thermogravimetric curves of onion skin powder of fifteen cultivars exploring gradual mass loss: 1, Agrifound Dark Red; 2, Agrifound Light Red; 3, Arka Kirthiman; 4, Bhima Kiran; 5, Bhima Shakti; 6, Bhima Shubhra; 7, Hissar-2; 8, Hissar-3; 9, NHRDF Red; 10, Phursungi Local; 11, Pusa Madhavi; 12, Pusa Red; 13, Pusa Riddhi; 14, Sukhsagar; 15, Udaipur Local.

TABLE 3: Maximum mass loss (with respect to the temperature) and residual mass of onion skin powder of fifteen cultivars.

Cultivar	Maximum mass loss (%)	Temperature range (°C)	Residual mass (%) at 299°C
Agrifound Dark Red	12.80 ± 0.05 <sup>b</sup>	220–270	70.98 ± 0.04 <sup>m</sup>
Agrifound Light Red	12.25 ± 0.08 <sup>b</sup>	240–290	50.90 ± 0.06 <sup>f</sup>
Arka Kirthiman	17.16 ± 0.07 <sup>ef</sup>	220–270	31.19 ± 0.02 <sup>c</sup>
Bhima Kiran	13.14 ± 0.05 <sup>c</sup>	220–270	63.75 ± 0.05 <sup>j</sup>
Bhima Shakti	12.55 ± 0.09 <sup>b</sup>	220–270	65.22 ± 0.05 <sup>k</sup>
Bhima Shubhra	16.47 ± 0.03 <sup>e</sup>	220–270	40.68 ± 0.04 <sup>d</sup>
Hissar-2	11.07 ± 0.02 <sup>a</sup>	220–270	67.73 ± 0.07 <sup>l</sup>
Hissar-3	22.16 ± 0.05 <sup>g</sup>	220–270	41.70 ± 0.02 <sup>d</sup>
NHRDF Red	15.27 ± 0.04 <sup>d</sup>	220–270	57.24 ± 0.02 <sup>h</sup>
Phursungi Local	25.31 ± 0.10 <sup>h</sup>	220–270	1.91 ± 0.08 <sup>a</sup>
Pusa Madhavi	11.32 ± 0.06 <sup>ab</sup>	250–299	59.56 ± 0.03 <sup>i</sup>
Pusa Red	22.72 ± 0.04 <sup>g</sup>	220–270	28.70 ± 0.04 <sup>b</sup>
Pusa Riddhi	13.08 ± 0.05 <sup>c</sup>	220–270	48.28 ± 0.01 <sup>e</sup>
Sukhsagar	17.02 ± 0.02 <sup>ef</sup>	220–270	52.04 ± 0.03 <sup>fg</sup>
Udaipur Local	10.67 ± 0.06 <sup>a</sup>	220–270	63.95 ± 0.04 <sup>j</sup>

Data are mean value ± standard deviation ( $n = 3$ ). Values in the same column with different letters are significantly different ( $P \leq 0.05$ ) by Duncan's multiple range test.

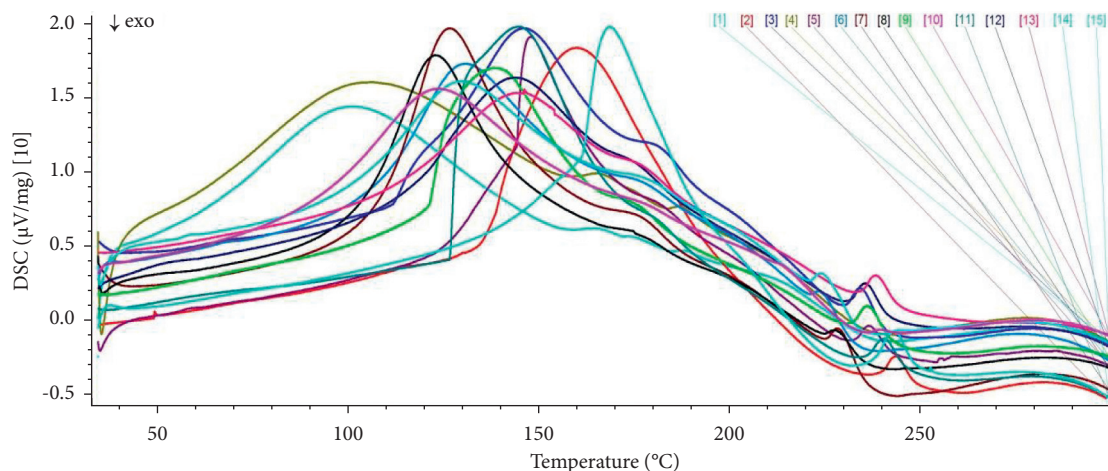


FIGURE 4: Combined differential scanning calorimetry curve of onion skin powder of fifteen cultivars: 1, Agrifound Dark Red; 2, Agrifound Light Red; 3, Arka Kirthiman; 4, Bhima Kiran; 5, Bhima Shakti; 6, Bhima Shubhra; 7, Hissar-2; 8, Hissar-3; 9, NHRDF Red; 10, Phursungi Local; 11, Pusa Madhavi; 12, Pusa Red; 13, Pusa Riddhi; 14, Sukhsagar; 15, Udaipur Local.

TABLE 4: Glass transition temperature (T<sub>g</sub>) of onion skin powder of fifteen cultivars.

Cultivar	Glass transition (T <sub>g</sub> ) temperature (onset °C-end °C)	Temperature of first endothermic peak (°C)	Temperature of second endothermic peak (°C)
Agrifound Dark Red	64.4–90.6	102.1 ± 0.02 <sup>a</sup>	—
Agrifound Light Red	135.9–140.9	160.4 ± 0.04 <sup>j</sup>	244.0 ± 0.09 <sup>h</sup>
Arka Kirthiman	111.1–132.7	146.5 ± 0.03 <sup>h</sup>	234.5 ± 0.10 <sup>c</sup>
Bhima Kiran	65.7–106.9	106.7 ± 0.05 <sup>b</sup>	—
Bhima Shakti	143.3–144.5	148.4 ± 0.05 <sup>i</sup>	238.5 ± 0.08 <sup>e</sup>
Bhima Shubhra	107.5–127.3	131.0 ± 0.03 <sup>e</sup>	223.7 ± 0.11 <sup>a</sup>
Hissar-2	107.0–110.2	127.0 ± 0.06 <sup>d</sup>	228.8 ± 0.13 <sup>b</sup>
Hissar-3	103.6–114.4	123.3 ± 0.04 <sup>c</sup>	228.1 ± 0.09 <sup>b</sup>
NHRDF Red	120.2–124.5	138.7 ± 0.06 <sup>f</sup>	236.4 ± 0.14 <sup>d</sup>
Phursungi Local	94.2–118.9	124.0 ± 0.02 <sup>c</sup>	—
Pusa Madhavi	127.2–128.6	144.7 ± 0.04 <sup>g</sup>	240.7 ± 0.08 <sup>f</sup>
Pusa Red	114.6–131.9	144.3 ± 0.03 <sup>g</sup>	236.0 ± 0.11 <sup>d</sup>
Pusa Riddhi	146.4–146.8	146.6 ± 0.02 <sup>h</sup>	238.8 ± 0.16 <sup>e</sup>
Sukhsagar	158.2–159.8	169.0 ± 0.03 <sup>k</sup>	243.7 ± 0.12 <sup>g</sup>
Udaipur Local	101.7–128.1	130.5 ± 0.06 <sup>e</sup>	224.1 ± 0.09 <sup>a</sup>

“—” no peak found. Data are mean value ± standard deviation ( $n = 3$ ). Values in the same column with different letters are significantly different ( $p \leq 0.05$ ) by Duncan's multiple range test.

one huge and second small endothermic peaks occurred at 138.7 and 236.4°C, respectively.

The study of cv. “Pusa Madhavi” revealed that the T<sub>g</sub> was taken place between the temperature range of 127.2°C–128.6°C. Two endothermic peaks were also obtained at different temperatures, one huge peak at 144.7°C and the second tiny peak at 240.7°C. Moderate ranges of T<sub>g</sub> (phase changing), that is, 111.1°C–132.7°C, 107.5°C–127.3°C, 107.0°C–110.2°C, 103.6°C–114.4°C, and 101.7°C–128.1°C, were obtained, respectively, for “Arka Kirthiman,” “Bhima Shubhra,” “Hissar-2,” “Hissar-3,” and “Udaipur Local” cultivars and a significant ( $p \leq 0.05$ ) difference was observed between cultivars (Table 4). Possible occurred reactions were dehydroxylation, decarboxylation, and demethoxylation [39, 40]. As per the study, the T<sub>g</sub> temperature revealed that the earliest phase change was taken place in cv. “Agrifound Dark Red” between 64.4°C and 90.6°C, while late phase change was confirmed in cv. “Sukhsagar” between 158.2°C and 159.8°C. Likewise, de Oliveira Cartaxo-Furtado et al. [38] studied the extract of *Syzygium cumini* L. leaves and found the largest endothermic peak between the temperature range of 114.2°C–150.69°C. Similarly, Da Silva et al. [41] observed a thermal event at 120°C during the analysis of freeze-dried camu camu fruit pulp (*Myrciaria dubia*). Apart from this, many endothermic peaks were obtained between 126.14°C and 325.50°C in the analysis of the dried extract of *Schinopsis brasiliensis* Engl. [30].

#### 4. Conclusions

In the present study, the skins of fifteen onion cultivars were investigated for various important parameters. The physicochemical analysis explored that “NHRDF Red” has the highest protein, fiber, and ash content among the cultivars studied, whereas “Pusa Riddhi” and “Pusa Red” were revealed as the best source of carbohydrates and fat among all the cultivars. Additionally, sulphur and mineral

analysis showed OSP as a rich source of essential minerals. TGA studies confirmed that cv. “Pusa Madhavi” was the most thermally stable, while “Pusa Red” was reported as the least thermally stable compare to others and DSC confirmed the earliest phase change in cv. “Agrifound Dark Red” but late phase change in cv. “Sukhsagar.” FTIR analysis suggested that all the cultivars were rich in various functional groups, such as methylene in the saturated aliphatic group, aromatic ring, carbohydrate, and sulphur oxy compounds. Onion skin waste in processing industries may be utilized for various purposes like functional/nutraceutical food development, energy, and biogas production [5–7, 10].

#### Data Availability

All the data pertaining to this work have been provided within the text.

#### Conflicts of Interest

The authors declare no conflicts of interest with respect to this work.

#### Authors' Contributions

Narashans Alok Sagar and Sunil Pareek conceived the experiment(s); Narashans Alok Sagar contributed to investigation; Vikas contributed to data analysis and PCA application; Narashans Alok Sagar and Ayon Tarafdar were responsible for writing of the original draft; Sunil Pareek contributed to reviewing and editing, supervision, and project administration; Anil Khar contributed to resources and draft editing.

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## Research Article

# Evaluation of Chemical, Functional, Spectral, and Thermal Characteristics of *Sargassum wightii* and *Ulva rigida* from Indian Coast

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Usage of seaweeds as a functional food/food ingredient is very limited due to paucity of scientific information about variations in the nutritional composition of seaweeds under diverse climatic conditions. *Sargassum wightii* and *Ulva rigida* seaweeds are found abundantly on the Southern Indian coastline and were thoroughly evaluated in this work. Crude fiber and lipid of *S. wightii* were higher ( $24.93 \pm 0.23\%$  and  $3.09 \pm 0.41\%$ , respectively) as compared to *U. rigida*; however, *U. rigida* had higher crude protein content ( $27.11 \pm 0.62\%$ ). Evaluation of mineral and CHNS content indicated that the concentration of potassium, magnesium, and calcium was  $1.36 \pm 0.08$  mg/g,  $8.39 \pm 0.80$  mg/g, and  $14.03 \pm 3.46$  mg/g, respectively, that was higher in the *S. wightii*, whereas *U. rigida* contained higher value of iron, carbon, and sulphur ( $0.70 \pm 0.13$  mg/g,  $37.72 \pm 4.63\%$ , and  $2.61 \pm 0.16\%$ , respectively). Swelling capacity ( $19.42 \pm 0.00$  mL/g DW to  $22.66 \pm 0.00$  mL/g DW), water-holding capacity ( $6.15 \pm 0.08$  g/g DW to  $6.38 \pm 0.14$  g/g DW), and oil-holding capacity ( $2.96 \pm 0.13$  g/g DW) of *U. rigida* were significantly ( $p < 0.05$ ) higher as compared to *S. wightii*. It was observed from DSC thermograms that *S. wightii* can be safely processed for food formulations even at a temperature of  $134^\circ\text{C}$ . The thermograms also revealed changes in the sulphated polysaccharide (fucoidan) profile due to the presence of hydroxyl and carboxyl groups with denaturation of proteins. TGA of *S. wightii* and *U. rigida* showed degradation temperature within the range of  $200\text{--}300^\circ\text{C}$  due to divergent polysaccharide compositions. FTIR spectroscopy suggested the presence of phenolic groups in both seaweeds (at  $1219\text{ cm}^{-1}$ ). Results of the study suggested that the manufacturing of functional food products from seaweeds could be beneficial and may aid in social upliftment of cultivators/fishermen.

## 1. Introduction

Seaweeds are potential source of bioactive compounds, phytochemicals, polysaccharides, dietary fibre,  $\omega$ -3 fatty acids, essential amino acids, vitamins, and minerals such as calcium, potassium, sodium, and phosphorous [1]. Bioactive compounds extracted from seaweeds have many therapeutic

properties such as antioxidant, anti-inflammatory, and antimicrobial activity [2]. The proximate composition, nutrients, and bioactive compounds present in seaweeds depend on several factors such as species, oxygen concentration, salinity of water, climatic season, intensity of UV radiation, and area of production [3, 4]. Seaweeds growing under such harsh conditions generate several

secondary metabolites that prevent their structural damage [5]. Commercially, seaweeds are utilized for extraction of stabilizers, thickening agents, agar, and gelling agents with diverse industrial applications.

Usage of seaweeds in Oriental and some European populations is significant, but not so among the Indian population. Countries like Japan, Malaysia, and France have legalized seaweeds as vegetables and condiments [6]. Indian coastal region is a rich source of seaweeds. Southern and South-eastern coast (Mandapam, Palk of Bay) of India are an industrial hub for seaweed cultivation, harvesting, and processing. Several species of brown, green, and red seaweeds are cultivated, as well as found, abundantly. Some genera of edible seaweeds among these are consumed by the coastal population but not by majority of the mainland people. Also, use of edible seaweeds in processed form is very scarce in India owing to palatability issues and unavailability of scientific data. Seaweeds therefore can be processed to bring them into palatable form, fit for various food applications considering their abundant availability, and miniscule consumption in the Indian diet. Moreover, due to growing consumer demand for nutritious food, seaweed can be easily popularized within the masses after establishing a strong scientific backbone to its merits.

Among brown seaweeds, *Sargassum* is an important genus. It is a good source of carbohydrate, minerals, proteins, essential amino acids (e.g., arginine, tryptophan, and phenylalanine),  $\beta$ -carotene, and vitamins [7]. Indian coast has more than 56 *Sargassum* species, and some species such as *Sargassum siliquosum* are utilized by local populations as an ingredient in salad, fish soup, and rice dishes [8]. Among *Sargassum*, *S. wightii* grows abundantly throughout the year on both eastern and western coasts. Significant variations in the nutritional composition of *S. wightii* have been reported with respect to seasonality and vegetative parts [7]. However, there are not many reports about its spectral and thermal characteristics and its relation with proximate, nutritional composition and functional properties. The present study is an attempt to elucidate these characteristics of *S. wightii*.

Same is the case of green seaweed (Chlorophyta). More than 43 genera of Chlorophyta have been recognized at the Indian coastal region [8]. In the Chlorophyta, *Ulva* is cultivated majorly. *Ulva* has various therapeutic properties due to the presence of ulvan, a sulfated polysaccharide that has antioxidant, antitumor, anticoagulant, immunomodulatory, wound dressing, tissue healing, and heavy metals binding ability [9, 10]. Among *Ulva*, *Ulva rigida* is a green macroalgae widely distributed in tropical oceans and Indian coastline. It is widely distributed in the Mandapam and adjacent areas of Tamil Nadu, south-eastern and Gujarat coast of India, respectively [7]. *Ulva* species is consumed in raw form or in soup preparations among the coastal residents [11].

Despite our best efforts, we could not find a study wherein thermal and spectral properties of these seaweeds are reported, and data regarding these properties shall help in processing of these seaweeds to formulate various

processed food products, as well as functional foods/nutraceuticals. However, ulvan (sulfated polysaccharide) extracted from *Ulva lactuca* (Monastir-Tunisia Coast) was thermally characterized, and it was reported that these polysaccharides showed stability even at 180°C, while rhamnose sulphate and uronic acid functional groups were observed through infrared spectroscopy [12]. Thus, the aim of the present study was to catalogue the proximate composition, functional properties, and thermal and spectral attributes of *S. wightii* and *U. rigida* seaweeds. We believe that the results of this investigation will be of immense help to food processing and nutraceuticals industries in order to come up with processed products or functional foods, whereby seaweed consumption can be increased in mainstream population.

## 2. Materials and Methods

**2.1. Chemicals.** Petroleum ether, boric acid, sodium hydroxide, hydrochloric acid, and nitric acid were purchased from Thermo Fischer, India. Corn oil was procured from Sigma-Aldrich (Bangalore, India). All other solvents and chemicals used were of analytical grade.

**2.2. Seaweed Collection and Processing.** Green (*Ulva rigida*) and brown (*Sargassum wightii*) seaweeds were obtained from the seaweed traders from Kanyakumari (8°05'02"N 77°32'46"E) Tamil Nadu, India, and Mandapam (9°17' N and 79°11' E), Tamil Nadu, India, in September 2018 with the help of scientists of the research institute, CSIR-Central Salt and Marine Chemicals Research Institute, Mandapam, India. Collected seaweed was cleaned with tap water to remove epiphytes, sand, and debris and then shade-dried at room temperature up to a total moisture content of  $21.53 \pm 0.05\%$  (wet basis). The shade-dried seaweeds were ground to powder using a mixer-grinder and passed through an 850 micron screen. The dried seaweed powder was stored at  $-20^\circ\text{C}$  in air tight bags for further analysis.

**2.3. Estimation of Chemical Composition of Seaweeds.** Proximate composition including total carbohydrates, crude fat, crude protein, crude fiber, total ash, and total moisture content of seaweeds was determined according to AOAC [13]. The values are reported in % dry weight (DW) basis.

### 2.4. Functional Properties of Seaweeds

**2.4.1. Swelling Capacity (SWC).** Swelling capacity of *U. rigida* and *S. wightii* was measured by bed volume technique [6, 14]. Briefly, 200 mg of dried seaweed powder was taken and mixed with 20 mL of deionized water and stirred vigorously. The effect of temperature on SWC was measured by keeping tubes at 25°C and 37°C for 24 h. SWC of the seaweeds was calculated using the following formula:

$$\begin{aligned} \text{SWC} &= \text{initial volume of water (mL)} \\ &- \text{volume of water after incubation (mL)}. \end{aligned} \quad (1)$$

The swelling volume was expressed as mL of swollen sample per gram of sample dry weight.

**2.4.2. Water Holding Capacity (WHC).** Water holding capacity of both seaweeds was measured by a modified centrifugation method [6, 14]. Briefly, 200 mg of dried seaweed powder was taken and mixed with 20 mL deionized water in the centrifuge tube. The tubes were kept in an incubator shaker (New Brunswick Scientific, Eppendorf AG, Germany) at 25°C and 37°C for 24 h. Sample was centrifuged (Sigma 3-18KS, Germany) at 14000 g at 37°C for 30 min, and supernatant was discarded. The wet weight of *U. rigida* and *S. wightii* was noted. Samples were then kept in an oven at 120°C for 2 h, and their dry weight was taken. WHC of *U. rigida* and *S. wightii* was calculated using the following formula:

$$\begin{aligned} \text{WHC} &= \text{wet weight of the sample (g)} \\ &- \text{dry weight of the sample (g)}. \end{aligned} \quad (2)$$

WHC was expressed as weight in grams of water held by 1 g of dried sample.

**2.4.3. Oil Holding Capacity (OHC).** According to the method of Wong and Cheung [6], OHC of both seaweeds was measured. Briefly, 3 g of dried seaweed powder was mixed with 10.5 g of corn oil in a centrifuge tube. Tubes were kept in the shaker for 30 min at room temperature followed by centrifugation at 2500 g for 30 min, and oil supernatant was collected. OHC was calculated using the following formula:

$$\begin{aligned} \text{OHC} &= \text{initial volume of oil (g)} \\ &- \text{volume of oil after incubation (g)}. \end{aligned} \quad (3)$$

The OHC was expressed as number of grams of oil held by 1 g of dried seaweed.

**2.5. Determination of Minerals in Seaweeds.** Samples were prepared using wet digestion method [15] with minor modifications. Briefly, 0.4–0.5 g sample was taken in a 50 mL beaker. To this, 1 mL of hydrogen peroxide was added for oxidation. Sample was digested using 5 mL concentrated nitric acid (69%) with constant heating at 70°C in a water bath. Digestion was continued for 1.5–2 h till attainment of a pale yellow color. Digested sample was transferred to a 50 mL volumetric flask, and the volume was adjusted using Milli-Q water. Metal composition in seaweeds was determined using an ICP-OES (Optima 7000 DV, Perkin Elmer). A multielement standard was used for analysis of minerals. The equipment was calibrated using different concentrations of the standard (5, 10, 25, 50, and 100 ppb) in 5% HNO<sub>3</sub>. Reference blank was taken as diluted (5%) HNO<sub>3</sub>. Analysis was carried out in triplicates. Results were expressed in mg/kg of seaweeds.

**2.5.1. Estimation of Elemental Content of Seaweeds Using the CHNS Analyzer.** Carbon, hydrogen, nitrogen, and sulphur content in the shade-dried seaweed samples were determined using the CHNS/O elemental analyzer (Euro EA Elemental Analyzer, Germany).

**2.6. Determination of Thermal Properties.** Thermal properties were analyzed using a differential scanning calorimeter (DSC) (NETZSCH, Germany) and thermogravimetric analyzer (TGA) (NETZSCH, Germany). Both experiments were performed under a nitrogen atmosphere. For TGA test, 5–10 mg of the test sample was placed into an aluminum pan, and a scan was performed under a temperature range of 20 to 600°C. The heating rate was kept at 10°C/min. For DSC analysis, approximately 5–10 mg of shade-dried seaweed powder was taken in a DSC aluminum pan and hermetically sealed using a lid. The sealed pan was loaded into the equipment at room temperature. An empty pan was used as reference. Flow rate of nitrogen was adjusted at 60 and 40 mL/min in purge lines 1 and 2, respectively. Heating was linearly ramped from 30°C to 250°C at a rate of 10°C/min.

**2.7. Fourier Transform Infrared (FTIR) Spectroscopy.** Shade-dried seaweed powder was analyzed using Fourier transform infrared (FTIR) spectrometer (Alpha Bruker, USA) at the wavenumber range of 4000–600 cm<sup>-1</sup> with resolution of 4 cm<sup>-1</sup> and 22 spectral scans. Samples were analyzed by Attenuated Total Reflectance (ATR) technology using a ZnSe crystal, and the spectrum of each sample was normalized with the background measurement. Smoothing of the sample spectra was done using Opus computer software.

**2.8. Statistical Analysis.** All measurements were carried out in triplicate (three separate lots of each seaweed species), and the values were reported as mean ± standard deviation. Data were analyzed with independent sample *t* test using SPSS statistical software package v.20 (IBM, USA) at 5% level of significance ( $p < 0.05$ ).

### 3. Results and Discussion

**3.1. Chemical Composition.** Table 1 shows proximate composition of brown (*S. wightii*) and green (*U. rigida*) seaweed. Crude protein content of *S. wightii* (6.43% DW) and *U. rigida* (27.11% DW) was found to be within the range of that reported for brown (3–15% DW) [7] and green (10–47% DW) seaweeds [6], respectively. Also, Balar et al. [16] reported protein content of *U. rigida* collected from Indian coastline in the range of 4.14–26.0% DW. Crude protein of *U. rigida* showed significantly ( $p < 0.05$ ) higher value as compared to *S. wightii*. However, the crude protein of the *S. wightii* was found to be lower than that of other *Sargassum* species (8–16.9% DW) [7]. Higher protein in green seaweeds is in agreement with previous reports [17]. Furthermore, the crude protein content of *S. wightii* and *U. rigida* was almost comparable to the same species found in the Saurashtra coast

TABLE 1: Proximate composition (%) of *S. wightii* and *U. rigida*.

Composition (% DW)	<i>S. wightii</i>	<i>U. rigida</i>
Crude protein	6.43 ± 0.39	27.11 ± 0.62*
Total ash	19.87 ± 0.34	19.63 ± 0.63
Crude fibre	24.93 ± 0.23*	18.65 ± 0.78
Total carbohydrate	45.66 ± 0.50*	31.87 ± 0.26
Crude lipid	3.09 ± 0.41	2.71 ± 0.70
Total moisture	21.33 ± 0.05	22.61 ± 0.80

Results are expressed as mean ± (n = 3). Values bearing \* are significantly different ( $p < 0.05$ ) from the corresponding column/seaweed in an independent sample *t* test. DW, dry weight.

(Western Indian coast: 8% DW) and in the Portuguese coast (29.5% DW), respectively [7, 18]. Kasimala et al. [4] reported similar results of protein in brown seaweed, *S. subrepandum* (6.93%) collected from Eritrean red sea coast of Gurgussum and Hirgigo bay. Variations in the crude protein content were reported to be proportional to thallus maturation of the *S. wightii* with higher crude protein content in the winter season (January–March) at the time of their developing phase, and lower protein content has been reported in the months of July to September on the southern Indian coast [7]. Thus, crude protein content found in our *S. wightii* correlates well with the results of Kumar et al. [7]. Similar range of protein content in seaweeds was also reported by Rohani-Ghadikolaei et al. [19]. Protein content has been reported to vary with type of species and seasonal variations [6, 20].

Total ash content of *S. wightii* and *U. rigida* is observed as 19.87% DW and 19.63% DW, respectively, which was comparable to the reports of Wong and Cheung [6]. Total ash content of *U. rigida* was slightly lower than other *Ulva* species such as *U. lactuca* and *U. pertusa* [1].

Crude fiber content of *S. wightii* (24.93% DW) was significantly ( $p < 0.05$ ) higher than *U. rigida* (18.65% DW) (Table 1). Crude fiber of *S. wightii* was found to be higher than the total dietary fiber of same species of *Sargassum*, while *U. rigida* showed lower value of crude fiber than other *Ulva* species [21]. Seaweeds are rich in dietary fibers. Soluble fraction of dietary fibers exhibits important functional properties such as antimutagenic, antioxidant, and anticoagulant [6, 22]. However, insoluble fibers and their physiological effects need much attention for further analysis.

In the present study, total carbohydrate content of *S. wightii* (45.66%) is significantly ( $p < 0.05$ ) higher than *U. rigida* (31.87%) (Table 1). Kumar et al. [7] reported 48.9–57.2% total carbohydrate content in the thallus portion of the *S. wightii* (Western Indian coast), which was comparable to the present study and 33.5% in *S. polycystum*, 18% in *S. myriocystum*, while other species of *Sargassum* were reported to have higher carbohydrate content such as *S. thunbergii* (67.2%) (Jeju Do Island, Korea) and *S. vulgare* (67.8%) (Buzios beach in the Northwest of Brazil). Balar et al. [16] reported 16.63–65.93% DW carbohydrate content in Indian seaweed *U. rigida*. Total carbohydrate content is related to the soluble and insoluble carbohydrate content of the *S. wightii* (Tamil Nadu, India) and highest values found in March and lowest in July [7].

Lipid content of seaweeds generally ranges from 1 to 3% [6]. In the present study, crude lipid content of *S. wightii* and *U. rigida* was found to be 3.09% and 2.71%, respectively (Table 1). However, lipid content of same species of *Sargassum* was comparable, ranging from 2 to 3%, and other species of *Sargassum* were found to be less than 1%; e.g., 0.3% in *S. thunbergii*, 3.8% in *S. echinocarpum*, and 2.0% in *S. ilicifolium* [7,19]. Also, same species of *U. rigida* from Portuguese coast showed similar result of lipid content (2%). Lipid content of other species of *Ulva* was also comparable to *U. fasciata* (1.83%), *U. reticulata* (2.03%), and *U. lactuca* (3.6%) [19, 23]. Wong and Cheung [6] reported 14.6% lipid content of *Ulva lactuca* that was lower from the *U. rigida*, whereas Balbar et al. [16] reported 0.8–3.1% lipid content in *U. rigida* collected from Indian coastal line. Lipid content of the *Sargassum* differs with seasonal variation (least increased from July to March) and the species of the seaweed. Young blades and thallus portion of *S. wightii* were reported to have highest and least amounts of lipids, respectively [7].

Moisture is a quality factor in the preservation of food products and affects stability of the food materials [21]. The total moisture content of *S. wightii* (21.22%) and *U. rigida* was observed to be 21.22% and 22.61%, respectively. Syad et al. [21] reported similar value of moisture content of same species of *Sargassum* (22.4%). Wong & Cheung [6] reported lower value 10.6% of *U. lactuca* than *U. rigida*.

**3.2. Functional Properties.** Figure 1 illustrates functional properties (SWC, WHC, and OHC) of *S. wightii* and *U. rigida*. SWC and WHC of *S. wightii* and *U. rigida* increased with the increase in temperature. At 25°C, SWC of *S. wightii* was 12.71 ± 0 mL/g dry weight (DW), which increased to 15.89 ± 0 mL/g DW at 37°C. Similarly, WHC of *S. wightii* increased from 5.83 ± 0.04 g/g DW (at 25°C) to 5.92 ± 0.14 g/g DW (at 37°C).

In case of *U. rigida*, the SWC and WHC were 19.42 ± 0.00 mL/g DW and 6.15 ± 0.08 g/g DW, respectively, at 25°C, which increased slightly to 22.66 ± 00 mL/g DW and 6.38 ± 0.14 g/g DW. The value of WHC at 25°C and 37°C of *U. rigida* is significantly ( $p < 0.05$ ) higher as compared to *S. wightii*. These values are higher than those reported for *S. wightii* (Gulf of Mannar, India) and *U. lactuca* (A Ma Wan, Hong Kong) [6, 14, 22].

WHC of *S. wightii* and *U. rigida* was lower than that reported for *G. edulis*, *Hypnea japonica*, *Hypnea charoides*, *U. lactuca*, *Laminaria*, and *Wakame* at 37°C. WHC value of both seaweeds at similar temperature was higher than the WHC of *S. wightii* (5.72 ± 0.14 g/g DW), *Fucus* (5.48 ± 0.42 g/g DW), and *Nori* (5.12 ± 0.15 g/g DW), which showed comparable results [22, 24].

SWC and WHC of seaweeds depend largely on the amount of protein and total dietary fibers present in their composition [6]. Also, lesser particle size with more surface area indicates higher WHC [25]. Both SWC and WHC directly affect the texture, mouthfeel, and freshness of the food products. Therefore, these help in avoiding loss of moisture from the products during processing [26]. In this study, SWC and WHC values were observed to increase with

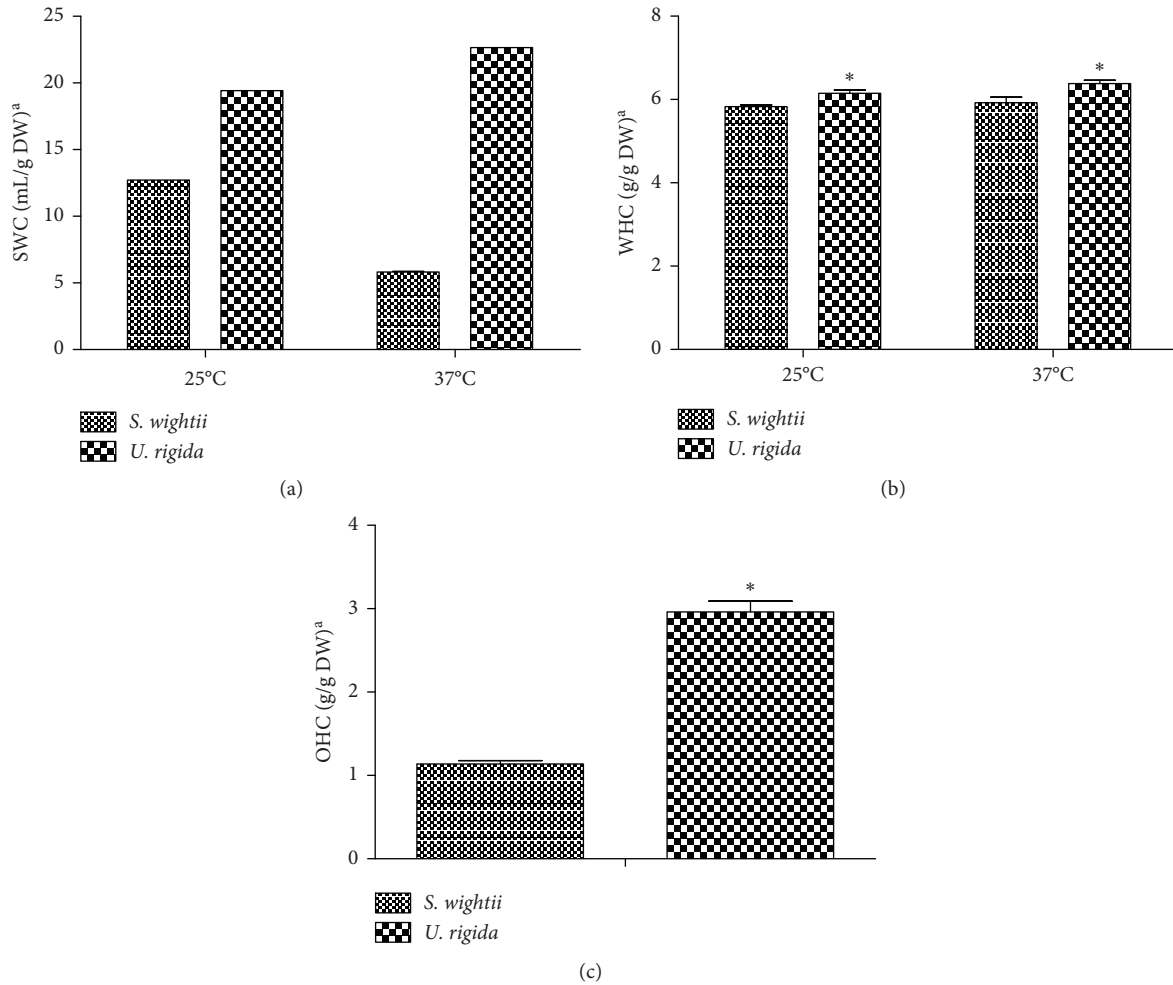


FIGURE 1: Functional properties of seaweeds *S. wightii* and *U. rigida*. <sup>a</sup>Results are expressed as mean  $\pm$  SD ( $n=3$ ). Values bearing \* are significantly different ( $p < 0.05$ ) from the corresponding seaweed in an independent sample  $t$  test.

temperature, which may be due to the increased solubility of fibers and proteins that contribute to the functional behavior [21]. Among the two seaweeds, *U. rigida* showed higher SWC and WHC, which shows its potential for use as functional ingredient to modify texture and viscosity, avoid syneresis, and reduce calories [6].

OHC value of *U. rigida* ( $2.96 \pm 0.13$  g/g DW) was found to be significantly ( $p < 0.05$ ) higher than *S. wightii* ( $1.14 \pm 0.04$  g/g DW) (Table 2). Comparable results were reported for *Laminaria*, *Nori*, and *G. edulis*, while it was higher than that of *H. japonica*, *H. charoides*, and *U. lactuca* [14, 22, 24]. According to Wong and Cheung [6], the hydrophobicity of proteins helps in fat absorption. The mechanism of OHC is mainly due to physical entrapment of oil by capillary attraction. Therefore, different proportions of polar side chains of amino acids present on the surface of protein molecules are responsible for variation in OHC of seaweeds. OHC of seaweeds is also related to hydrophilic nature, overall charge density, and particle size of the individual particles.

TABLE 2: Mineral composition of *S. wightii* and *U. rigida* analyzed by ICP-OES.

Name of the element	Observed concentration (mg/g)	
	<i>S. wightii</i>	<i>U. rigida</i>
Mg	$8.39 \pm 0.80^*$	$6.07 \pm 0.49$
P	$0.32 \pm 0.00$	$0.44 \pm 0.01^*$
K	$1.36 \pm 0.08^*$	$0.27 \pm 0.03$
Ca	$14.03 \pm 3.46^*$	$2.39 \pm 0.17$
Fe	$0.30 \pm 0.03$	$0.70 \pm 0.13^*$
Cr	$0.002 \pm 0.00$	$0.003 \pm 0.00$
Mn	$0.008 \pm 0.00$	$0.02 \pm 0.00^*$
Cu	$0.007 \pm 0.00$	$0.02 \pm 0.01$

Results are expressed as mean  $\pm$  ( $n=3$ ). Values bearing \* are significantly different ( $p < 0.05$ ) from the corresponding column/seaweed tested in an independent sample  $t$  test.

**3.3. Mineral Content.** Table 2 shows mineral and trace element analysis of *S. wightii* and *U. rigida*. Analysis revealed higher potassium ( $1.36 \pm 0.08$ ) and calcium ( $14.03 \pm 3.46$ )

content in *S. wightii* as compared to *U. rigida*. Potassium plays an important role in electrical conductivity and functioning of brain [21], whereas seaweed-sourced calcium (calcium carbonate) has been reported to be utilized more effectively as compared to cow milk's calcium [23]. *U. rigida* showed higher magnesium in comparison to *S. wightii* contain. Magnesium plays an important role in functioning of central nervous system and also helps in eliminating the symptoms of Parkinson's and Alzheimer's disease [21]. In addition to macrominerals, both the seaweeds contained trace elements, which also play a major function in the human body. Iron is one of the important trace elements, being a major component of hemoglobin. Iron content in *S. wightii* was found to be lower (0.30 mg/g) as compared to *U. rigida* (0.70 mg/g). Apart from iron, *S. wightii* and *U. rigida* had trace elements like chromium, manganese, and copper. Elemental bioaccumulation by seaweeds is affected by season, thallus age, pH, habitat, and exposure to residential and industrial effluents [23]. In the present study, *S. wightii* showed lower amounts of potassium, calcium, magnesium, iron, and copper as compared to that reported by Murugaiyan and Sivakumar [22] and Syad et al. [21] for *S. wightii* collected from Gulf of Mannar, India. This variation could be due to season, time of collection, climatic factors, etc. *Ulva rigida* showed slightly lower amounts of potassium, calcium, magnesium, iron, manganese, and copper as compared to the same species of *Ulva* obtained from northwest Iberian, Spain and Portuguese coast [24]. The results of mineral analysis hold significance for use of both seaweeds for nutraceuticals (dietary supplements functional foods industry). Also, Soares et al. [27] reported that subcritical water extracts of *Saccorhiza polyschides* (brown seaweed) are rich in minerals (Na, S, Ca, and Mg) that can be used in the development of fertilization products.

**3.4. Elemental (CHNS) Composition.** Table 3 shows carbon, hydrogen, nitrogen, and sulphur percentage of *S. wightii* and *U. rigida*. Nitrogen, sulphur, and hydrogen content of *U. rigida* was higher than that of *S. wightii*, while carbon content of *S. wightii* was found to be higher than *U. rigida*. Sulphur content of seaweeds represents amount of sulphur binds with the polysaccharides to form sulfated polysaccharides such as fucoidan in *S. wightii* and ulvan in *U. rigida*. These sulfated polysaccharides play critical role in free radical scavenging and show substantial antioxidant activity [12].

**3.5. Decomposition and Glass Transition Temperature of Seaweeds.** Thermogravimetric analysis (TGA) curves for *S. wightii* and *U. rigida* indicate the initial weight loss by water evaporation in the range of 0–100°C (*U. rigida* showed loss at 63.3°C) [28]. The decomposition of *S. wightii* and *U. rigida* occurred in the temperature range of 200–300°C, positioned at 282.4°C and 273.2°C, respectively (Figure 2(a)). Besides, *S. wightii* showed its second decomposition at 533.3°C. Chemical decomposition of organic material initiates at a temperature range of 200–300°C. At temperature ranges of 220–260°C and 315–390°C, decomposition of

TABLE 3: CHNS elemental analysis of *S. wightii* and *U. rigida*.

Seaweed	N %	C %	H %	S %
<i>S. wightii</i>	1.02 ± 0.06	33.16 ± 4.89	5.62 ± 0.79	0.70 ± 0.06
<i>U. rigida</i>	4.33 ± 0.09	37.72 ± 4.63	6.94 ± 0.82	2.61 ± 0.16

Results are expressed as mean ± SD ( $n=3$ ).

hemicellulose and cellulose, respectively, takes place [29]. For D-arabinose and D-mannose, pyrolysis reaction occurred at a temperature range of 120–310°C and for D-xylose within the range of 140–310°C [29], and final degradation above 500°C is associated with burning of carbonaceous residues triggered by intense heating [30].

Thermograms of the seaweed powder (*S. wightii* and *U. rigida*) showed a characteristic endothermic peak indicating their respective glass transition temperatures ( $T_g$ ) and enthalpy. *S. wightii* and *U. rigida* exhibited a  $T_g$  of 134.7°C and 78.1 °C, respectively (Figure 2(b)). Glass transition temperature of *U. rigida* was found to be lower than *S. wightii*. This could be due to inter- and intramolecular hydrogen bonding formed by the polysaccharide hydroxyl and carboxylate groups [18]. Here, the lower  $T_g$  value of *U. rigida* may be due to higher concentrations of hydroxyl groups of polysaccharides. Rodriguez-Jasso et al. [20] reported that fucoidan extract of *Fucus vesiculosus* shows weight loss due to dehydration at a temperature range of 25°C and 110°C. They also reported that, at 120°C, pyrolysis of polysaccharide extract can take place, which could lead to phase transition.

Narrowing of the peaks was observed in both seaweed powders. The area under these peaks was used to determine the enthalpy of both seaweeds. Enthalpy of *S. wightii* and *U. rigida* was calculated as 224.484 J/g and 127.030 J/g, respectively. Increased enthalpy of *S. wightii* indicates more energy required for peptide and glycosidic bond-breakage. Therefore, the net enthalpy indicates the effects of endothermic events (breakdown of hydrogen bonds) [31]. Thermal data of both seaweeds provide appropriate temperature intervals, so that they can be used in the formulation of seaweed-based food products such as noodles. Kumar et al. [31] reported that glass transition temperature ( $T_g$ ) of seaweed infused coffee varied from 121.3°C to 139.3°C that changes the flavor of seaweed infused coffee with increase in  $T_g$ . Therefore, only a specific range of temperature (<200°C) is appropriate for product formulation using seaweed, beyond which degradation could take place.

**3.6. Functional Group Analysis.** Table 4 shows the infrared spectrum analysis of the seaweeds in the form of % absorbance versus wave number ( $\nu$ ). *S. wightii* and *U. rigida* showed weak absorption peak above 3600  $\text{cm}^{-1}$  which may be due to the presence of O-H stretching bend, which shows specific pattern of hydrated inorganic compounds [31]. Strong absorption peak was observed above 3200  $\text{cm}^{-1}$  in *U. rigida*, while *S. wightii* showed weak absorption peak corresponding to N-H and O-H stretching possibly due to the presence of polysaccharides and amino acids, native to



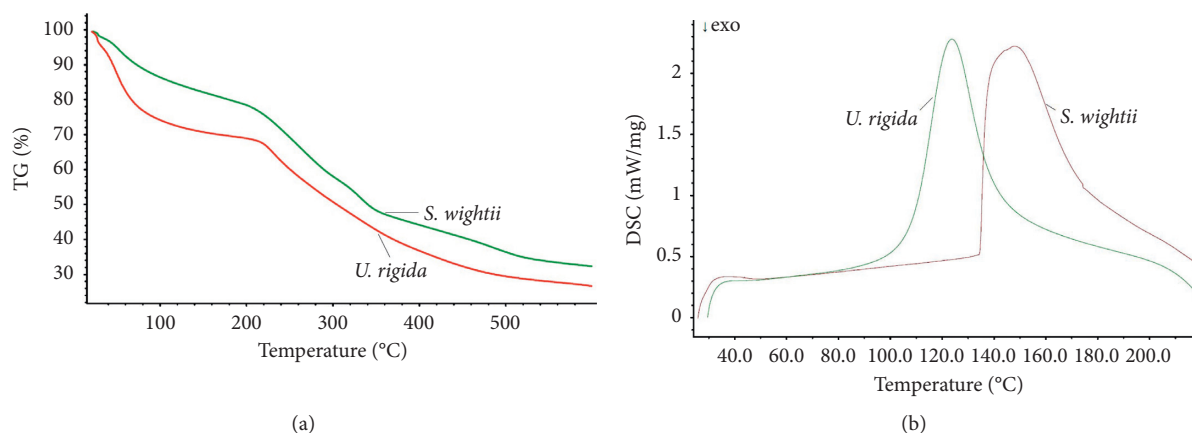


FIGURE 2: Thermograms of *S. wightii* and *U. rigida* from (a) TGA (20°C to 600°C) and (b) DSC (25°C to 250°C).

TABLE 4: FTIR absorption frequencies and functional group of *Sargassum wightii* and *Ulva rigida*.

Wavenumber		Functional group	Compounds	References
<i>S. wightii</i>	<i>U. rigida</i>			
3859	—			[31]
3740	3750	OH-stretching	Alcohol	[31]
3669	—			—
—	3245			—
2372	—	C-O stretching	Phosphorous	[10]
1684	1631	C=O stretching, N=O asymmetric stretching	Ester, pectin, amide	[32]
1527	1527			C=C stretching
—	1417	C-O stretching, O-H bending	Cutin	[33]
—	1219	S=O stretching, C-O stretching	Sulphates, phenols	[33]
1023	1023	S=O stretching	Starch and polysaccharides	[33]
—	842	C-H bending		Glucose, galactose
699	666	C-S stretching, C=S stretching	Sulphates	[33]

seaweeds [31]. Weak absorption peak was observed above  $2300\text{ cm}^{-1}$  in both seaweeds, which shows presence of C-O stretching. Strong absorption peak was observed above  $1600\text{ cm}^{-1}$  indicating C=O, N=O stretching in both seaweeds suggesting a sign of ester and amide groups responsible for characteristic flavor of seaweeds [10, 32]. Peaks from  $1400\text{--}1500\text{ cm}^{-1}$  and  $1500\text{--}1600\text{ cm}^{-1}$  indicate several modes of C=C, C-O, O-H stretching, which may be due to the presence of lignin. Peaks above  $1200\text{ cm}^{-1}$  and  $650\text{ cm}^{-1}$  in both seaweeds indicate S=O, C-O, C-S, and C=S stretching resulting from the sulphate and phenols present in the seaweeds. Strong absorption peak at  $1023\text{ cm}^{-1}$  in both seaweed points to S=O stretching indicating the presence of starch and polysaccharides. Weak absorption peak above  $800\text{ cm}^{-1}$  in *U. rigida* corresponds to C-H stretching, which may be due to the presence of glucose and galactose [33]. Peaks above  $1200\text{ cm}^{-1}$  in both seaweeds represent the phenol groups that indicate that both seaweeds are flourish in antioxidants, which can be utilized in development of functional food and their extract may be characterized for the bioactive compounds which can be useful in nutraceuticals formulation. It has also been reported that marine macroalgae produces wide variety of volatile organic compounds such as hydrocarbons, terpenes,

phenols, alcohols, aldehydes, ketones, esters, fatty acids, and halogen or sulfur-containing compounds, which are responsible for its characteristic flavor and freshness, and these volatile compounds have been well identified by Spanish researchers in *U. rigida* [34].

#### 4. Conclusions

The outcome of the present study is that we have very well documented proximate composition, few nutritional attributes, chemico-functional properties, and thermal as well as spectral characteristics of seaweeds, *S. wightii* and *U. rigida*, collected in the month of September 2018 from the Southern coast of India. Both seaweeds were found to be a good source of nutrients such as crude protein, crude fiber, calcium, iron, and sulfur. The three functional properties, viz., SWC, WHC, and OHC, were found to have direct relation with the fiber and proteins in the seaweeds. Also, it was observed that *S. wightii* can be safely processed up to  $134^\circ\text{C}$  as seen from the DSC thermograms without any major phase change phenomenon, whereas TGA results revealed that decomposition of organic material takes place within a temperature range of  $200\text{--}300^\circ\text{C}$  for both seaweeds. Therefore, both seaweeds may be utilized to develop fiber rich food products/

as an ingredient in products withstanding this much temperature. Moreover, FTIR spectra showed phenolic groups in both seaweeds, suggesting that they can act as potential antioxidants ultimately to be used for the development of functional foods and nutraceuticals. This seems to be the first report mentioning thermal (DSC and TGA) and spectral (FTIR) attributes of these seaweeds. We believe that large scale use of these seaweeds by food industry could raise socioeconomic status of the cultivators/fishermen. Nevertheless, future work shall be directed towards *in vivo* studies to establish bioavailability of nutrients and polyphenols present in these seaweeds so as to get comprehensive and conclusive data for their pertinent exploitation.

### Data Availability

All data pertaining to this work are included within this article.

### Conflicts of Interest

The authors declare no conflicts of interest.

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