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# Formulation of cookies enriched with spent Heiyai (*Elaeagnus latifolia* L.) obtained from supercritical fluid extraction and their quality analysis

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The utilization of spent samples from supercritical fluid extraction (SFE), which are rich in minerals and crude fibers, offers a sustainable method for enhancing food products. This study investigates the potential of utilizing spent Heiyai (*Elaeagnus latifolia* L.) obtained from the SFE process to produce cookies with improved nutritional value and sensory attributes. Cookies were prepared by substituting wheat flour with SFE-obtained spent Heiyai (SH) at five different percentages: 0%, 2%, 5%, 10%, and 15% (w/w). The proximate and biochemical compositions, such as total phenolic content (TPC), antioxidant activity, and mineral content, were determined. The sensory scores of various cookie samples were evaluated using a fuzzy logic approach, and the samples were given ranks based on their sensory qualities. The study observed that the proximate compositions of the cookies were increased with increasing SH percentage, and the samples were comparable to the control (HC1), except for protein content, where the control had a higher level. The TPC, antioxidant activity, and mineral content in the cookies significantly increased with the addition of SH. The Heiyai cookies were ranked according to the highest similarity values for the five samples: HC3 (very good) > HC2 (very good) > HC1 (good) > HC5 (good) > HC4 (fair). The fuzzy logic analysis indicated that the overall ranking of quality criteria was followed as texture > taste > appearance > colour > flavor.

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## Sustainability spotlight

Spent Heiyai (*Elaeagnus latifolia* L.) obtained after supercritical fluid extraction (SFE) is free from chemical residues as SFE is a green technology. Spent Heiyai is a good source of iron, magnesium, zinc, potassium and other minerals along with fiber. Our work utilises spent Heiyai obtained from SFE in the formulation of cookies enriched with fiber and minerals. The present approach reduces waste while helping in the development of functional food products to support the sustainable development goal 3.

## 1. Introduction

The semi-wild edible berry Heiyai (local name in Manipur) or silverberry or oleaster in India is an underutilized fruit belonging to the Elaeagnaceae family. Heiyai fruit is available in the hills and valleys of Manipur, India.<sup>1</sup> Consuming fruit and vegetables is highly beneficial for our health as they are abundant in minerals, vitamins, and bioactive substances. Fruits that belong to the category of underutilized fruits have therapeutic and pharmacological qualities and significant concentrations of vital nutrients that support overall health and well-being. These fruits provide diverse types of vitamins and minerals crucial for maintaining good health.<sup>2</sup> The Heiyai fruit has numerous proven medical benefits. It is used in the traditional Turkish, Jordanian, and Iranian folk medicines for treating various health conditions, serving as a diuretic, tonic,

antipyretic, and antidiarrheal agent and to treat kidney disorders, diarrhoea, dysentery, tetanus, rheumatoid arthritis, and asthma.<sup>3</sup> Numerous health-beneficial components can be found in oleaster (another name of Heiyai) such as minerals, like magnesium, calcium, potassium, iron, phosphorus, copper, and vital fatty acids, including palmitic, linoleic, and palmitoleic acids. Carotenoids and various bioactive substances such as saponin, cardiac glycoside, alkaloids, triterpenoids, terpenes, coumarins, phytoenes, tannins, amino acids, and polysaccharides are also found in it.<sup>4</sup> These fruits offer significant health benefits that are currently unknown to the public due to unawareness and limited accessibility. The nutritional, functional, and sensory attributes of the mesocarp layer of the oleaster fruit, which is used as a flour in many product categories, including biscuits, yogurt, breakfast cereals, gluten-free cakes, and bread, have been studied.<sup>5</sup>

Supercritical fluid extraction (SFE) was used to extract lycopene from Heiyai, and the characterization of lycopene was conducted in the study by Devi *et al.*<sup>5</sup> As a continuation of the

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previous study, the current research utilized spent Heiyai from the extraction process to develop a value-added product. SFE is an effective and environmentally friendly extraction technique that separates valuable bioactive molecules from natural sources using supercritical fluids, typically carbon dioxide. This method produces significant SH along with the extracts. SH is rich in proteins, fibers, fats, and carbohydrates as well as bioactive substances such as vitamins and antioxidants. Due to its nutritional composition, SH is appropriate for various applications, including the development of new functional foods and nutraceuticals.

Several studies have shown the potential for using spent materials from SFE in product development. The spent material from the SFE extraction of used coffee grounds is a valuable source of fiber and is enriched with polysaccharides, including galactomannans and arabinogalactans. Their composition of caffeic acid and chlorogenic acid contributes to their potent hypotensive and antioxidant properties. Furthermore, the low glycemic index of these dietary fibers facilitates weight loss and aids in preventing disorders such as type 2 diabetes, which are associated with obesity.<sup>6</sup> In another study by Ghosh *et al.*,<sup>7</sup> the spent material from supercritical walnut kernel extraction was incorporated into cookie recipes instead of wheat flour as a valorization strategy. The utilization of the spent sample after the SFE process of Heiyai offers a sustainable method for developing cookies with wheat flour. This approach not only aims to minimize waste but also seeks to repurpose the spent samples, thereby enhancing the efficiency of the extraction process and promoting sustainable manufacturing practices. The dried oleaster fruit, which is a variety of Heiyai fruit, can be supplemented to flour in baking. This flour can also be incorporated into the production of functional products such as ice cream, yogurt, infant foods, and confections.<sup>3</sup>

The current study also investigates the potential of utilizing SH from the SFE process to produce cookies with improved nutritional value and sensory attributes. Fuzzy logic is a valuable method for analyzing imprecise data and making significant conclusions about the acceptance, rejection, and ranking as well as the strong and weak qualities of food.<sup>8</sup> In fuzzy modeling, relationships between independent (colour, flavor, texture, and overall acceptance) and dependent (ranking, acceptance, rejection, and the strong and weak attributes of the sample) variables are developed using linguistic variables (example: not satisfactory, good, and excellent).<sup>1</sup>

An experimental sensory evaluation analyzes and assesses a product's sensory characteristics, which can be perceived through sight, smell, touch, taste, and hearing. Human perception is imprecise, but assessments based on language provide a realistic evaluation from the evaluator's perspective.<sup>9</sup> Fuzzy logic plays a crucial role in analyzing imprecise and uncertain data. Fuzzy logic is based on fuzzy set theory and contains infinite truth values.<sup>10</sup>

This study demonstrates an effective approach to managing spent sample utilization. Cookies were formulated using SH with significant functional and nutritional components, such as fibers and minerals, after the supercritical fluid extraction process. This study focuses on assessing the biochemical

characteristics of the developed cookies incorporated with SH. The SH powder has a floury texture, distinct flavor, and functional characteristics; it is a rich source of minerals, dietary fibers, and bioactive substances. However, there has not been any study reported on using SH flour in the preparation of cookies. Fuzzy logic was employed for both the sensory evaluation and ranking of the cookies according to their qualitative attributes. The strongest and weakest characteristics of the developed cookies were also identified.

## 2. Material and methods

### 2.1. Materials

Heiyai (*Elaeagnus latifolia* L.) was procured from March to April from Imphal, Manipur, India. The proximate composition (% wet basis) of Heiyai was moisture ( $81.04\% \pm 0.34\%$ ), ash ( $3.10\% \pm 0.26\%$ ), crude fat ( $0.60\% \pm 0.72\%$ ), crude fiber ( $7.08\% \pm 0.15\%$ ), crude protein ( $1.31\% \pm 0.11\%$ ), and total carbohydrate ( $6.91\% \pm 1.89\%$ ). After the supercritical extraction process, SH was collected from the SFE system and stored at  $-20\text{ }^{\circ}\text{C}$  until further use.

### 2.2. Preparation of heiyai cookies

The cookie dough was prepared using the standard ratio of 3 : 2 : 1 of flour to fat to sugar, and the specific ingredients are listed in Table 1. In a separate bowl, butter was beaten thoroughly using a beater, and then, all the ingredients were gradually added and mixed thoroughly. SH was substituted with wheat flour at five different percentages: 0%, 2%, 5%, 10%, and 15% (w/w). The selected substitution levels were based on preliminary trials aimed at identifying suitable incorporation ranges that have no adverse effects on dough handling or sensory characteristics. The cookie sample prepared without SH (0%) was kept as a control sample. The dough was then refrigerated for 30 min to maintain the shape of the cookies during baking. A cookie cutter was used to cut the cookies, placed in a preheated baking oven (SM-502) at  $180\text{ }^{\circ}\text{C}$  and baked for 30 min. After baking, the cookies were cooled on the baking sheet for 30 min before being transferred to an airtight container for further analysis.

### 2.3. Quality attributes of the prepared cookies

**2.3.1. Proximate composition.** The proximate composition of the prepared Heiyai cookies was determined using the AOAC (2020) method.<sup>11</sup> Precisely, the moisture content was measured utilizing a hot air oven at  $105\text{ }^{\circ}\text{C}$ . The ash content was estimated using a muffle furnace set at  $550\text{ }^{\circ}\text{C}$  for 6 h. The crude fiber content was estimated using a sequence of acid and alkaline hydrolysis methods. The protein and fat contents were determined using the Kjeldahl method and Soxhlet apparatus, respectively. To determine the carbohydrate content of the cookies, the moisture, ash, crude fiber, fat, and protein values were subtracted from 100. Each result was obtained in duplicate.

**2.3.2. Extraction method for assessing TPC and antioxidant activity.** A Heiyai cookie extract was obtained using 80%



Table 1 Ingredients used for the preparation of cookies

Sample	Wheat flour (g)	SH (%)	Butter (g)	Sugar (g)	Milk (mL)	Baking powder (g)
HC1	200	0	130	60	7	3
HC2	196	2	130	60	7	3
HC3	190	5	130	60	7	3
HC4	180	10	130	60	7	3
HC5	170	15	130	60	7	3

methanol as the solvent with slight modifications to the method mentioned by Hussain *et al.*<sup>12</sup> In summary, 100 mL of 80% methanol was used to soak 0.5 g of each cookie sample. The mixture was continuously stirred at 300 rpm for 24 h at room temperature using an orbital shaker. Afterward, the mixture was filtered, and the resulting solution was evaporated at 45 °C

using a vacuum evaporator. For further analysis, the extract was stored at 4 °C.

**2.3.2.1 Total phenolic content (TPC).** The TPC of the Heiyai cookie extract was estimated using a method mentioned by Hussain *et al.*<sup>12</sup> About 500  $\mu$ L of each extract was taken, and 0.5 mL of Folin–Ciocalteu reagent diluted in distilled water (1 :

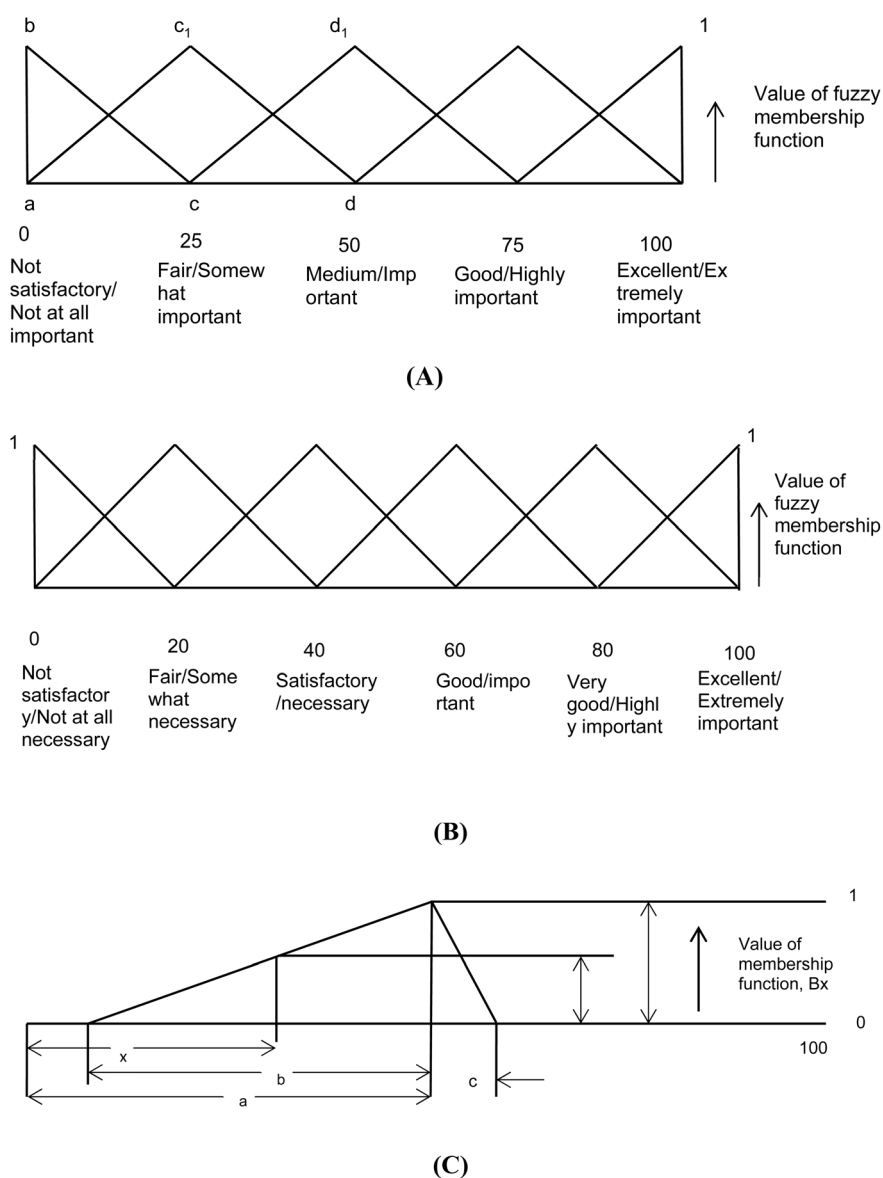


Fig. 1 Representation of the triangular membership function distribution pattern of (A) sensory scale, (B) standard fuzzy scale and (C) graphical view of one of the overall sensory scores as a triplet (*a*, *b* and *c*) (adapted and modified from: Swami Hulle (2015)<sup>16</sup> and Das (2005)<sup>17</sup>).



2) was added to this. 2 mL of 7% Na<sub>2</sub>CO<sub>3</sub> was added to this mixture and incubated for 30 min. Absorbance was measured at 760 nm using a spectrophotometer, with methanol (80%) used as a blank solution. TPC was expressed as the milligram of gallic equivalent (GAE) per g of the cookie extract.

**2.3.2.2 DPPH free-radical scavenging activity assay.** About 500 µL of the Heiyai cookie extract was mixed with 3 mL of prepared DPPH solution in a test tube and incubated in the dark for 15 min. Absorbance was measured at 517 nm, and 80% methanol was used as a blank sample.<sup>13</sup> The DPPH free radical scavenging activity was determined using eqn (1).

$$\text{Free radical scavenging activity(\%)} = \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \times 100, \quad (1)$$

where  $A_{\text{control}}$  = absorbance of the control sample and  $A_{\text{sample}}$  = absorbance of the control sample.

**2.3.3. Mineral analysis.** About 0.5 g of Heiyai cookie powder was placed in a Kjeldahl tube, and 20 mL of a solution of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and nitric acid (HNO<sub>3</sub>) in 3 : 1 ratio was added to the tube. The digestion process was performed for approximately 4 h at 420 °C, which was continued until a clear solution appeared. Then, the mixture was allowed to cool and filtered using a syringe filter (Whatman Uniflo™) with a pore size of 0.2 µm. Once the filtration was completed, the mineral analysis was conducted using ICP-OES. Results are expressed in mg kg<sup>-1</sup>.

**2.3.4. Colour analysis.** The colour values of the Heiyai cookies were analyzed using a colorimeter (Ultrascan VIS, Hunterlab, USA). The colorimeter was calibrated using a white plate. It measures two coordinates,  $a^*$  and  $b^*$ , as well as the brightness ( $L^*$ ) value. The  $L^*$  value falls between 0 (absolute black) and 100 (absolute white). Negative  $a^*$  values indicate the presence of green, while positive  $a^*$  values indicate the presence of red. The  $b^*$  value indicates the presence of yellowness for positive values and blueness for negative values.

**2.3.5. FTIR measurements.** The Heiyai cookie powder samples were subjected to FTIR analysis using a method described by Singhal *et al.* (2024).<sup>14</sup> The dried sample was mixed with potassium bromate and hydraulic pressed to form a pellet. The pellet was scanned using an FTIR spectrophotometer (Nicolet Impact 410 FTIR, Thermo Fisher Scientific, USA) at a frequency range of 400–4000 cm<sup>-1</sup>. The measurements were done in the transmittance mode.

**2.3.6. Sensory evaluation.** A total of 30 semi-trained panellists conducted the sensory evaluation of the Heiyai cookies.

trained about the different quality attributes of the cookies and different terms used in the sensory scale (ASTM STP-758, 1981).<sup>15</sup> Using fuzzy logic sensory scales, the panellists assessed the samples based on five specific quality attributes: colour, flavour, taste, texture, and appearance. Five different concentrations of cookies were coded as HC1, HC2, HC3, HC4, and HC5. Panellists were instructed to rate the samples as 'poor', 'fair', 'medium', 'good', and 'excellent' for assessments of quality attributes and assign each quality attribute of cookies in general in terms of a tick mark against the appropriate choices.

The sensory evaluation used a five-point language scale to gather responses. In Fig. 1(A), the numerical values of 0, 25, 50, 75, and 100 correspond to the following sensory evaluation ratings: not satisfactory, fair, medium, good, and excellent, respectively. Quality attributes were rated using the following categories: not at all important (NI), somewhat important (SI), important (I), very important (VI), and extremely important (EI). The panellists also offered sensory evaluation ratings based on a scoring methodology using a score chart. Each sample was assigned a random two-digit code for identification. MATLAB R2017b (The MathWorks Inc.) was employed to analyse the fuzzy logic using the language data that was recorded during the evaluation process. The sensory scores of the Heiyai cookies were converted into a triangular membership distribution, known as triplets, representing the sensory scale, as shown in Fig. 1.

The fuzzy modelling of sensory evaluation involves several key steps: (a) determining the overall sensory scores for cookies samples using triplets, (b) membership function calculations on a standard fuzzy scale, (c) calculating the overall membership functions on a standard fuzzy scale for the Heiyai cookies, (d) estimating similarity values and ranking of the Heiyai cookies, (e) conducting a general ranking of the quality attributes of the Heiyai cookies, and the final step involves (f) ranking the quality attributes of the individual Heiyai cookies.<sup>18</sup>

Triplets corresponding to the sensory scale, judges' count for each sample of the Heiyai cookies, and the related summed sensory score were obtained from eqn (2) for each quality characteristic of Heiyai cookies.

where HC<sub>*i*</sub> denotes the sensory score triplet;  $c_1$ ,  $c_2$ ,  $c_3$ ,  $c_4$ , and  $c_5$  represent the scores provided by each judge; and  $i$  stands for samples 1 to 5.

The triplet consists of 3 numbers: the first number denotes the abscissa coordinate, the second number denotes the path from the first number to the left, and the third number

$$HC_i = \frac{c_1(0 \ 0 \ 25) + c_2(25 \ 25 \ 25) + c_3(50 \ 25 \ 25) + c_4(75 \ 25 \ 25) + c_5(100 \ 25 \ 0)}{c_1 + c_2 + c_3 + c_4 + c_5} \quad (2)$$

The panellists comprised both male and female participants aged above 18 and not having any medical/health conditions that impact their sensory judgement skills. The sensory panellists were selected based on the interests and were initially

represents the distance from the first number to the right. The second and third numbers of the triplets had a membership function value of zero, while the first number had a membership function value of one.<sup>19</sup> The triplets ( $a \ b \ c$ ) for each



triangular membership are displayed in Fig. 1(C), and the quality attribute triplet values of the Heiyai cookies was determined by eqn (2). In general, the corresponding ratio of each triplet to the maximum of the sum of the triplets was used to determine the triplet of relative weightage ( $W$ ) for the quality attributes. Using eqn (3), the overall sensory attribute ( $C_i$ ) for the sample of  $i$ th Heiyai cookies was calculated.

$$\text{OHC}_i = \sum_{k=1}^6 (\text{HC}_i \text{ for the } k^{\text{th}} \text{ attribute}) (W \text{ for the } k^{\text{th}} \text{ attribute}) \quad (3)$$

where  $k$  denotes the six sensory attributes examined.

The triplet ( $a \ b \ c$ ) was multiplied by the delete triplet ( $o \ p \ q$ ) using eqn (4), applying the triplet matrix multiplication rule.

$$(a \ b \ c) \times (o \ p \ q) = (a \times o + a \times p + o \times b + a \times q + o \times c) \quad (4)$$

For every standard fuzzy scale, a triangle distribution pattern was used to generate the membership function ( $F_1$ – $F_6$ ), as shown in eqn (5). The membership function has a maximum value of 1 and is composed of a set of ten numbers, as shown in Fig. 1(C).

Not satisfactory/not at all necessary  $F_1 = (1, 0.5, 0, 0, 0, 0, 0, 0, 0, 0)$

Fair/somewhat necessary  $F_2 = (0.5, 1, 1, 0.5, 0, 0, 0, 0, 0, 0)$

Satisfactory/necessary  $F_3 = (0, 0, 0.5, 1, 1, 0.5, 0, 0, 0, 0)$

Good/important  $F_4 = (0, 0, 0, 0, 0.5, 1, 1, 0.5, 0, 0)$

Very good/highly important  $F_5 = (0, 0, 0, 0, 0, 0, 0.5, 1, 1, 0.5)$

Excellent/extremely important  $F_6 = (0, 0, 0, 0, 0, 0, 0, 0, 0.5, 1)$  (5)

The value of membership function  $B_x$  for a given value of  $x$  on the abscissa can be expressed by eqn (6), which can be used to determine the value of membership function  $B_x$  at  $x = 0, 10, 20, 30, 40, 50, 60, 70, 80, 90$ , and 100 for each sample and its triplet.<sup>3</sup>

$$B_x = \frac{x - (a - b)}{q} \text{ for } (a - b) < x < l$$

$$B_x = \frac{((a + c) - x)}{n} \text{ for } l < x < (a + c) \quad (6)$$

$B_x = 0$  for all other values of  $x$ .

The  $B_x$  values for the cookies HC1, HC2, HC3, HC4, and HC5 were denoted as BHC1, BHC2, BHC3, BHC4, and BHC5, respectively. Using these values for each of the Heiyai cookie samples, the similarity value ( $S_m$ ) of any sample was determined using eqn (7).

$$S_m = \frac{F \times B'_x}{\text{Maximum of } (F \times F' \text{ and } B_x \times B'_x)} \quad (7)$$

where  $F'$  represents the transposed membership function ( $F$ ), and  $B'_x$  represents the transpose of the overall membership function  $B_x$ .

The selected quality attributes used to assess the Heiyai cookies were ranked based on their similarity values. Each of the five Heiyai cookie samples were analyzed using six  $S_m$  values, corresponding to the six levels of  $F$ , while the value of  $B_x$  remained constant for all the Heiyai cookie samples.

Similarly, the  $S_m$  values for each of the six sensory qualities were determined using a consistent approach. Panelists consistently showed a strong preference for samples with higher  $S_m$  values over those with lower  $S_m$  values.<sup>20</sup> Based on the  $S_m$  values, a similar process was used to rank the quality attributes of the Heiyai cookies.

**2.3.7. Statistical analysis.** The data are presented as mean  $\pm$  standard deviation, with each analysis conducted in triplicate. To determine if there were significant differences ( $p \leq 0.05$ ) among the samples, the analysis of variance (ANOVA) and Duncan's multiple range tests were performed using the IBM SPSS software.

## 3. Results and discussion

### 3.1. Proximate composition of the heiyai cookies

The proximate composition of the Heiyai cookies in five different formulations prepared using SFE-obtained SH is shown in Table 2. The moisture content of the Heiyai cookies ranged from 2.04% to 3.86%. It was observed that the moisture content significantly increased with the amount of SH added to the cookies. In a similar study by Ikuomola *et al.*,<sup>21</sup> the moisture level of cookie samples made from the blends of wheat flour and malted barley bran ranged from 3.34% to 4.6%. The study by Gernah *et al.*<sup>22</sup> revealed a similar pattern, with cookies in their study prepared from the wheat-brewer's spent grain flour blends having a greater moisture content (5.20% to 9.30%). High moisture content in baked goods, such as cakes, cookies, and bread, promotes the growth of bacteria, yeast, and mold, which can cause spoiling.<sup>23</sup> Additionally, the low moisture level (less than 10%) ensures high storage stability by preventing microbial spoilage.<sup>4</sup> The presence of high moisture in SH cookies are likely due to the hygroscopic nature of the residue powder, which absorbs moisture from the surroundings.

Similarly, the ash content of the Heiyai cookies increased from 1.36% to 2.78% while that of the control sample (HC1), prepared without SH, showed a decreasing trend. The increase in the ash content could be due to the presence of mineral-rich SH in the cookies. Agricultural byproducts like maize stalks and rice husks often enhance ash content due to their mineral makeup, which explains the lower ash concentration in the control sample. Overall, the ash content reflects the mineral contribution from added ingredients.<sup>24</sup> Results indicated that the substitution of SH at various levels significantly varied the fiber content ( $p < 0.05$ ) of the Heiyai cookies. The crude fiber content of the cookies ranged from 1.30% to 3.28%, with sample HC5 exhibiting the highest fiber content (3.28%) and HC1 the lowest (1.30%). The results indicated that increasing the concentration of SH led to a corresponding increase in the fiber content of the Heiyai



Table 2 Proximate composition of the Heiyai cookies<sup>a</sup>

Sample	Moisture	Ash	Crude fat	Crude fiber	Protein	Carbohydrate
HC1	2.04 ± 0.05 <sup>e</sup>	1.36 ± 0.01 <sup>e</sup>	30.31 ± 0.12 <sup>d</sup>	1.30 ± 0.06 <sup>e</sup>	12.76 ± 0.20 <sup>b</sup>	50.47 ± 0.26 <sup>e</sup>
HC2	2.54 ± 0.58 <sup>d</sup>	1.41 ± 0.09 <sup>d</sup>	29.05 ± 0.03 <sup>d</sup>	1.94 ± 0.07 <sup>d</sup>	12.27 ± 0.07 <sup>b</sup>	53.54 ± 0.30 <sup>d</sup>
HC3	3.05 ± 0.02 <sup>c</sup>	1.57 ± 0.01 <sup>c</sup>	25.70 ± 0.15 <sup>c</sup>	2.12 ± 0.8 <sup>c</sup>	11.64 ± 0.17 <sup>a</sup>	55.58 ± 0.24 <sup>c</sup>
HC4	3.55 ± 0.5 <sup>b</sup>	1.76 ± 0.08 <sup>b</sup>	23.29 ± 0.37 <sup>b</sup>	2.93 ± 0.02 <sup>b</sup>	8.55 ± 1.73 <sup>a</sup>	58.13 ± 0.11 <sup>b</sup>
HC5	3.86 ± 0.05 <sup>a</sup>	2.78 ± 0.01 <sup>a</sup>	22.81 ± 0.74 <sup>a</sup>	3.28 ± 0.10 <sup>a</sup>	8.71 ± 0.05 <sup>a</sup>	59.11 ± 0.10 <sup>a</sup>

<sup>a</sup> All values represent the mean ± standard deviation of three replicates. Samples exhibiting distinct superscripts within the same column are significantly different ( $p \leq 0.05$ ).

cookies. The control sample (HC1), consisting only the refined wheat flour, contained the lowest amount of crude fiber. This could be explained by the high crude fiber content of 5.9% found in Heiyai fruits.<sup>25</sup> In comparison, the other samples that included SH exhibited a higher concentration of crude fiber than the control sample (HC1).

The fat content of the cookies, as shown in Table 2, varied between 22.81% in the HC5 sample and 30.31% in the control sample (HC1). The control sample, which was baked with refined flour (*W*), had the highest fat content. In contrast, the samples baked with SH substitution exhibited lower fat amounts. Upon comparing all the samples containing SH, the fat contents of HC1 and HC5 were significantly different ( $p \leq 0.05$ ).

The amount of carbohydrate content in the cookies was generally high, ranging from 50.47% in the sample HC1 to 59.11% in the sample HC5. The cookies that contained the most SH had the highest carbohydrate percentage, indicating that the differences in carbohydrate content among the cookie samples were significant.

The Heiyai cookies had protein levels ranging from 8.71% to 12.76% (Table 2), and the values among the samples were significant ( $p \leq 0.05$ ). The control sample (HC1) had the highest protein content, while the sample HC5, which had a greater addition of SH and less wheat flour, exhibited the lowest protein content. The substitution of SH led to a decrease in the protein content of the samples.

The presence of acrylamide was assessed using a qualitative assessment using the FTIR spectra of the samples containing the highest and lowest concentration of SH. The observed peaks around 3236  $\text{cm}^{-1}$  correspond to N–H stretching vibrations, while the peaks near 2935  $\text{cm}^{-1}$  are attributed to C–H stretching. Additionally, a band observed at approximately 1475  $\text{cm}^{-1}$  may be associated with C–N stretching or CH bending vibrations. However, the absence of a prominent peak around 1660  $\text{cm}^{-1}$ , which is characteristic of the amide (C=O) group of acrylamides, suggests that acrylamide is either absent or present in negligible amounts in the samples. The FTIR spectra is included in the SI file (Fig. S2). However, due to spectral overlap in complex, heterogeneous matrices, confirmatory chromatographic analysis is required for better results and quantification.<sup>26</sup>

### 3.2. Total phenolic content (TPC) and DPPH free-radical scavenging activity of the heiyai cookies

Total phenolic content (TPC) is a key indicator of the antioxidant potential of functional food products. In this study, the

effect of incorporating SH on the TPC of cookies was evaluated. The cookie sample with the highest concentration of SH (HC5) had the highest total phenolic content ( $13.83 \pm 1.36$  mg GAE per g) while the control (HC1) had the lowest ( $1.52 \pm 0.48$  mg GAE per g). A consistent pattern was found, with phenolic content increasing in proportion to the amount of SH incorporated (Fig. 2). The phenolic compounds reported to be present in *E. latifolia* are gallic acid, *p*-coumaric, *m*-coumaric, ferulic acid, chlorogenic acid, caffeic acid, and catechin.<sup>27</sup>

The evaluation of the free-radical scavenging activity of the cookies formulated with varying concentrations of SH was conducted and compared with that of the control sample. The free radical scavenging activity (DPPH) was estimated using a method by Şahin (2023);<sup>28</sup> this study reported better results for similar cookies incorporated with *Elaeagnus* fruit pulp, and DPPH was found to be a better indicator of antioxidant activity in their study. The results are presented in Fig. 2. The free-radical scavenging activity ranged from 7.87% to 25.77%. The findings indicated that replacing SH resulted in a greater free-radical scavenging activity than the control sample (HC1). Additionally, the cookie (HC5) with 15% SH exhibited the highest antioxidant activity. These results indicated that the free-radical scavenging activity increased significantly with the addition of all the five levels of SH in the cookies.

Since the Heiyai fruit is a significant source of total phenolic content, adding it to the cookies increased their DPPH free-radical scavenging activity. Total phenolic content and free-radical scavenging activity are directly correlated. A similar

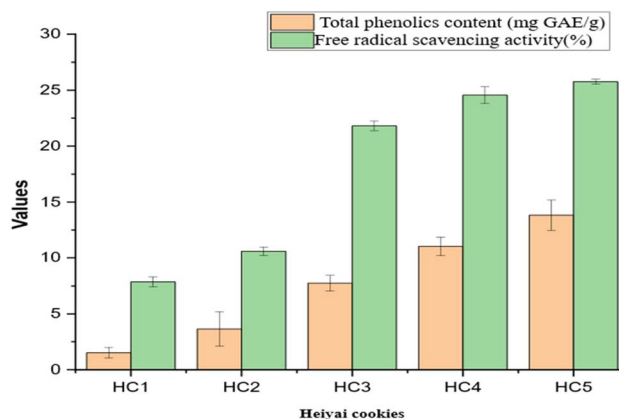


Fig. 2 Total phenolic content and free-radical scavenging activity of the Heiyai cookies. All data are expressed as mean ± SD of three replicates.



Table 3 Colour analysis of the Heiyai cookies

Sample	$L^*$	$a^*$	$b^*$
HC1	73.72 ± 1.42 <sup>a</sup>	2.367 ± 0.54 <sup>e</sup>	26.12 ± 1.62 <sup>d</sup>
HC2	69.31 ± 0.92 <sup>b</sup>	8.27 ± 0.42 <sup>d</sup>	29.50 ± 0.60 <sup>c</sup>
HC3	66.17 ± 0.08 <sup>c</sup>	12.67 ± 0.31 <sup>c</sup>	32.59 ± 0.57 <sup>b</sup>
HC4	61.65 ± 0.22 <sup>d</sup>	16.25 ± 0.07 <sup>a</sup>	54.77 ± 0.24 <sup>a</sup>
HC5	48.28 ± 0.12 <sup>e</sup>	14.06 ± 0.26 <sup>b</sup>	32.95 ± 0.60 <sup>b</sup>

pattern was found in the study conducted by Hussain *et al.*<sup>10</sup> There are limited studies available on the use of *Elaeagnus latifolia* L. and similar species in products like cookies. Şahin (2023)<sup>28</sup> formulated oleaster (*Elaeagnus angustifolia* L.) parts in cookies and reported similar observations like improved phenolic content and antioxidant capacity. The higher antioxidant activity may be attributed to the sample composition.

### 3.3. Colour analysis of the heiyai cookies

The colour parameters for the five cookies formulated with different concentrations of SH, each containing 0%, 2%, 5%, 10%, and 15% of SH, are shown in Table 3. The sample HC1 (0%) exhibited the highest  $L^*$  value among all the formulations. As the amount of SH increased, the cookies' lightness ( $L^*$ ) decreased, and thus, HC5 had the lowest  $L^*$  value. This means that the cookies became darker as the level of SFE-obtained SH increased. The colour from the SH may have contributed to the drop in the  $L^*$  value. When added at higher concentrations, this could be the reason for cookies becoming darker. Another reason might be the Maillard reaction, which is thought to produce brown pigments during baking by caramelizing the sugar present and causing Maillard browning.<sup>29</sup> On the other

hand, the redness value and the yellowness value of the Heiyai cookies, indicated by the  $a^*$  and  $b^*$  values, respectively, significantly ( $p < 0.05$ ) increased from the control sample (HC1), which was made without SH, to the cookies containing the highest level of SH (HC5). Overall, the colour data indicated that the cookies appeared significantly darker due to an increase in SH. The photographs of cookie samples incorporated with SH are available in the SI.

### 3.4. Mineral composition of the heiyai cookies

The mineral analysis was conducted on the five different cookies made with varying levels of SH. The findings are presented in Fig. 3. Five major minerals were analysed: Fe, Mg, Zn, K, and Na. The iron (Fe) content significantly increased from the control sample (HC1) to HC5, which contained the highest concentration of SH. There were substantial differences in the iron content among the cookie samples; HC5 had the highest concentration ( $0.684 \pm 0.03 \text{ mg kg}^{-1}$ ), while HC2 had the lowest ( $0.102 \pm 0.67 \text{ mg kg}^{-1}$ ). The sodium concentration varied from  $23.79 \pm 0.12 \text{ mg kg}^{-1}$  (HC2) to  $28.71 \pm 0.03 \text{ mg kg}^{-1}$  (HC5). HC5 had the highest potassium level at  $13.46 \pm 0.01 \text{ mg kg}^{-1}$  while HC2 had the lowest at  $5.965 \pm 4.016 \text{ mg kg}^{-1}$ . Compared to other samples, HC4 and HC5 exhibited significantly greater potassium concentrations, indicating that they may serve as good sources of potassium. Additionally, potassium was found to be the most abundant mineral in all the cookies, highlighting that these products contain a high amount of potassium. Magnesium levels varied less, ranging from  $1.547 \pm 0.21 \text{ mg kg}^{-1}$  (HC4) to  $1.265 \pm 0.54 \text{ mg kg}^{-1}$  (HC2). The cookie (HC4) had the highest magnesium level, even though the variation was minimal compared with the lowest value of HC2. Both HC1 and HC5 showed consistent zinc levels at  $0.208 \text{ mg kg}^{-1}$ , while HC3 exhibited the lowest zinc concentration

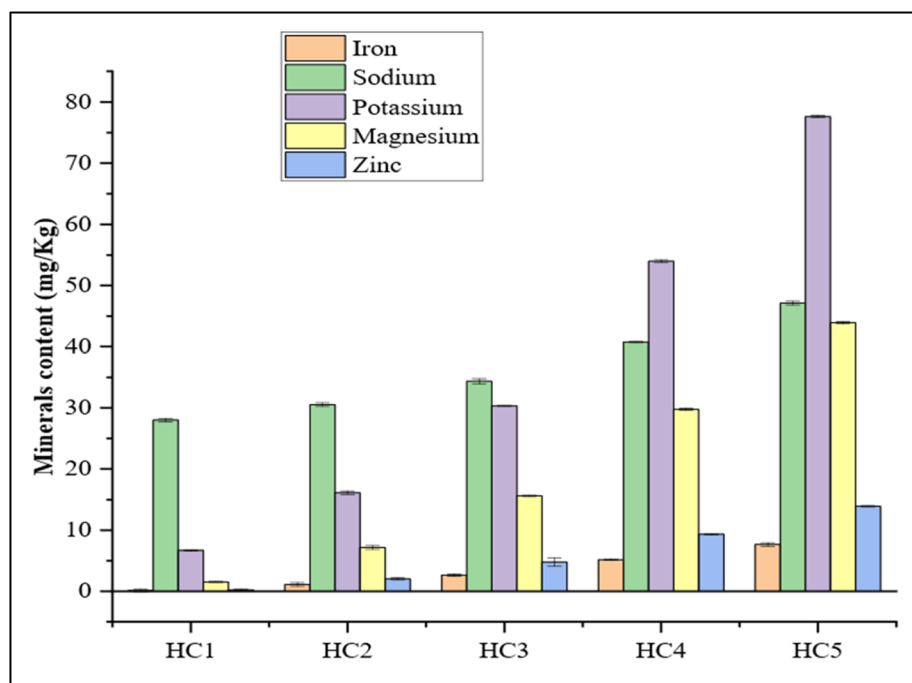


Fig. 3 Analysis of the mineral composition in the Heiyai cookies. All data are presented as mean ± standard deviation (SD) of three replicates.



Table 4 Similarity values and ranking of the quality attributes for each of the Heiyai cookies

Sensory scale	Colour	Flavour	Taste	Texture	Appearance
<b>HC1</b>					
Not satisfactory	0.024	0.026	0.029	0.001	0.020
Fair	0.278	0.294	0.306	0.142	0.252
Satisfactory	0.701	<b>0.731</b>	<b>0.736</b>	0.515	0.666
Good	<b>0.731</b>	0.704	0.698	<b>0.762</b>	<b>0.746</b>
Very good	0.281	0.221	0.209	0.498	0.335
Excellent	0.019	0.006	0.003	0.107	0.031
Rank	III	V	IV	I	II
<b>HC2</b>					
Not satisfactory	0.024	0.012	0.000	0.000	0.018
Fair	0.273	0.209	0.135	0.127	0.242
Satisfactory	0.687	0.614	0.509	0.488	0.650
Good	<b>0.726</b>	<b>0.770</b>	<b>0.769</b>	<b>0.753</b>	<b>0.749</b>
Very good	0.302	0.391	0.512	0.537	0.355
Excellent	0.026	0.042	0.108	0.134	0.037
Rank	V	I	II	III	IV
<b>HC3</b>					
Not satisfactory	0.026	0.004	0.000	0.000	0.026
Fair	0.293	0.156	0.117	0.117	0.288
Satisfactory	<b>0.730</b>	0.535	0.466	0.466	0.710
Good	0.707	<b>0.766</b>	<b>0.749</b>	<b>0.749</b>	<b>0.719</b>
Very good	0.224	0.460	0.567	0.567	0.262
Excellent	0.006	0.081	0.150	0.150	0.015
Rank	V	I	II	III	IV
<b>HC4</b>					
Not satisfactory	0.483	0.447	0.551	0.076	0.370
Fair	<b>0.954</b>	<b>0.970</b>	<b>0.906</b>	0.483	<b>0.970</b>
Satisfactory	0.371	0.420	0.278	<b>0.828</b>	0.526
Good	0.010	0.021	0.000	0.517	0.043
Very good	0.000	0.000	0.000	0.092	0.000
Excellent	0.000	0.000	0.000	0.000	0.000
<b>HC5</b>					
Not satisfactory	0.030	0.188	0.136	0.037	0.034
Fair	0.320	0.754	0.678	0.353	0.341
Satisfactory	<b>0.754</b>	<b>0.776</b>	<b>0.848</b>	<b>0.762</b>	<b>0.770</b>
Good	0.692	0.216	0.326	0.666	0.679
Very good	0.200	0.000	0.018	0.196	0.182
Excellent	0.001	0.000	0.000	0.003	0.000
Rank	V	II	I	IV	III

at  $0.150 \pm 0.65 \text{ mg kg}^{-1}$ . The majority of the minerals, including iron, sodium, potassium, and zinc, were consistently found in the highest concentrations in the HC5 sample. This suggests that HC5 may be the most nutrient-rich sample assessed. In contrast, HC2 may be less effective as a mineral source due to its comparatively low mineral composition. Variations in the addition of different amounts of SH may explain the differences in the mineral composition of the samples. The mineral content of the samples increased effectively as the concentration of SH increased. These results suggest that some samples have potential as functional foods with enhanced nutritional profiles.

### 3.5. Sensory evaluation of the heiyai cookies by fuzzy logic

A total of 30 panelists assessed the sensory qualities of the five different Heiyai cookies (HC1, HC2, HC3, HC4, and HC5),

Table 5 Similarity values of cookie samples and their ranking

Sensory scale	HC1	HC2	HC3	HC4	HC5
Not satisfactory	0.004	0.000	0.000	0.230	0.029
Fair	0.125	0.088	0.085	<b>0.798</b>	0.281
Satisfactory	0.423	0.351	0.348	0.749	0.655
Good	<b>0.680</b>	0.631	0.635	0.211	<b>0.696</b>
Very good	0.591	<b>0.649</b>	<b>0.658</b>	0.000	0.331
Excellent	0.195	0.246	0.249	0.000	0.040
Rank	III	II	I	V	IV

yielding significantly divergent evaluations. Sensory and quality attributes, in general, were utilised to calculate the triplet's correspondent with them based on eqn (2). To determine the relative weightage triplets, the triplets of sensory attributes were used, which were then multiplied with each sample's triplet using the multiplication rule given in eqn (4). The triplet acquired was the overall sensory score for every sample, as shown in Table 4 was determined using eqn (3).

The values of the membership function were calculated using the equations presented in eqn (5) and (6), and the six-point sensory scale was expressed as membership function values ( $F_1, F_2, F_3, F_4, F_5$ , and  $F_6$ ) on a standard fuzzy scale (eqn (5)). Similarity values and quality attributes in general for each Heiyai cookie sample were calculated, and all values are shown in Table 4, respectively.

**3.5.1. Ranking of the heiyai cookies according to the similarity values.** The similarity values of the five Heiyai cookies developed using different concentrations of SH are shown in Table 5. HC3, developed by incorporating 5% of SH, showed the highest similarity value of 0.658, categorizing it as 'very good'. The cookie sample HC2 was also placed in second rank with a similarity value of 0.649, falling into the 'very good' category and bearing significant similarity with HC2. The third preferred sample was HC1 (control sample) that obtained a similarity value of 0.680, placing it in the 'good' category. Both HC1 and HC5 were classified in the 'good' category. Lastly, the lowest similarity value was observed in HC4 at 0.289, positioning it in the 'fair' category. The Heiyai cookies were ranked based on the highest similarity values for the five samples. Results showed the following trend: HC3 (very good) > HC2 (very good) > HC1 (good) > HC5 (good) > HC4 (fair).

**3.5.2. Quality attributes in general and the ranking.** The quality attribute "texture" was ranked highest by panellists with a similarity value 0.966 in the 'highly important' category, based on the similarity values ranking procedure. Similar to the first preference, the second was classified as 'taste' with a similarity score of 0.855. 'appearance' came in third, followed by 'colour' and 'flavour', with similarity scores of 0.961, 0.832, and 0.805 under the same heading of 'important'. Based on the fuzzy logic analysis, the overall ranking of the quality criteria indicates that texture > taste > appearance > colour > flavour.

**3.5.3. Quality attributes for each of the heiyai cookies.** The quality attributes of each of the five Heiyai cookies were evaluated and ranked according to the fuzzy ranking rule (Table 4). After a thorough assessment by the panellists, it was determined that texture stood out as the most appealing attribute



among the five qualities assessed for the Heiyai cookies samples. Specifically, the texture of samples HC1, HC2, and HC3 was classified as “good”, with similarity values of 0.762, 0.753, and 0.741, respectively. Additionally, the quality attribute ‘texture’ was first preferred in HC1 and HC4. On the other hand, the samples HC4 and HC5 were rated to be in the “satisfactory” category, with similarity values of 0.828 and 0.762, respectively.

The samples HC2 and HC3 ranked first out of the five, with similarity values of 0.770 and 0.766, respectively, placing them in the “good” category for the quality attribute of taste. In the HC4 sample, the quality attribute ‘flavour’ was ranked the highest, with a similarity score of 0.766.

In the HC4 sample, the panelists indicated that they were less satisfied with the colour, flavour, taste, and appearance of the product. These attributes received similarity values of 0.954, 0.970, 0.906, and 0.970, respectively, falling into the “fair” category.

The quality attribute ‘texture’ was ranked the highest by panellists, with a similarity value 0.966 in the ‘highly important’ category, based on the similarity values ranking procedure. Similar to the first preference, the second was classified as ‘taste’ with a similarity score of 0.855. ‘Appearance’ came in third, followed by ‘colour’ and ‘flavour’, with similarity scores of 0.961, 0.832, and 0.805, respectively, under the same heading of ‘important’. Based on the fuzzy logic analysis, the overall ranking of the quality criteria indicates the trend of texture > taste > appearance > colour > flavour.

## 4. Conclusion

The present study demonstrates an approach to utilize the spent Heiyai material obtained after the super critical extraction process as a good source of dietary fibres and minerals. Further, a fuzzy logic-based approach for the effective representation and ranking of quality attributes was presented. A similar approach can be a useful tool for product development and the ranking of quality attributes in cookies. The study demonstrates similar sustainable approaches for using the spent raw material for value addition in food product formulations.

## Conflicts of interest

There are no conflicts to declare.

## Data availability

All data related to the study are included in this manuscript itself. However, for any clarification required, they can be supplied upon request.

Supplementary information (SI) is available. See DOI: <https://doi.org/10.1039/d5fb00634a>.

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