

# Sustainable Food Technology

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## Sustainability Spotlight

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The spent obtained after the supercritical fluid extraction(SFE) of Heiyai (*Elaeagnus latifolia* L.) is free from chemical residues, as SFE is a green technology. The spent is a good source of Iron, Magnesium, Zinc, Potassium and other minerals along with fiber. Our work utilises the spent obtained from SFE in formulation of cookies enriched with fiber and minerals. The present approach reduces at the same time used for development of functional food product to support Sustainable Development Goal 3.



## Formulation of cookies enriched with Heiyai spent (*Elaeagnus latifolia* L.) obtained from Supercritical Fluid Extraction and its quality analysis

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**Abstract:** The spent samples from Supercritical Fluid Extraction (SFE), rich in minerals and crude fibers, offer a sustainable method for enhancing food products. This study investigates the potential of utilizing Heiyai (*Elaeagnus latifolia* L.) spent from the SFE process to produce cookies with improved nutritional value and sensory attributes. Cookies were prepared by substituting wheat flour with SFE Heiyai spent (HS) at five different percentages: 0%, 2%, 5%, 10%, and 15% (w/w). The proximate and biochemical composition, such as total phenolic content (TPC), antioxidant activity, and mineral content, was 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 the percentage of HS, and the samples were comparable to the control (HC1), except for protein content, where the control had a higher level. The value of the TPC, antioxidant activity, and mineral content in the cookies significantly increased with the addition of HS. 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.

**Keywords:** *Supercritical fluid extraction, Heiyai spent, nutritional composition, minerals, fuzzy logic*

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### 1. Introduction



The semi-wild edible berry known as 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>6</sup>. Consuming fruit and vegetables is highly beneficial for our health as they are abundant in minerals, vitamins, and bioactive substances. Fruits like those that belong to 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>8</sup>. This fruit has numerous proven medical benefits. It is used in traditional Turkish, Jordanian, and Iranian folk medicine for various health conditions, including as a diuretic, tonic, antipyretic, antidiarrheal, and to treat kidney disorders, diarrhoea, dysentery, tetanus, rheumatoid arthritis, and asthma <sup>20</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. Carotenoids and various bioactive substances such as saponin, cardiac glycoside, alkaloid, triterpenoids, terpene, coumarin, phytoene, tannins, amino acid, and polysaccharides are also especially found in it <sup>15</sup>. These fruits have significant health benefits that are currently unknown to the public due to unawareness and limited accessibility. To increase the nutritional, functional, and sensory attributes of the mesocarp layer of oleaster fruit, which is used as flour in many product categories, including biscuits, yogurt, breakfast cereals, gluten-free cakes, and bread, have been studied <sup>20</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>7</sup>. As a continuation of the previous study, the current research utilized the 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. SFE primarily uses carbon dioxide and is an efficient and environmentally friendly process for extracting valuable compounds from various materials. This method produces significant HS along with the extracts. These HS are rich in proteins, fibers, fats, and carbohydrates, as well as bioactive substances such as vitamins and antioxidants. Due to their nutritional composition, HS are essential for various applications, including the improvement of new functional foods and the development of 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>21</sup>. In another study by Ghosh *et al.*<sup>10</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 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>20</sup>.

The current study also investigates the potential of utilizing HS 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>23</sup>. In fuzzy modeling, relationships between independent (colour, flavor, texture, overall acceptance, etc.) and dependent (ranking, acceptance, rejection, strong and weak of the sample attributes) variables are developed using linguistic variables (example: not satisfactory, good, excellent, etc.)<sup>6</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, so assessments based on language provide a realistic evaluation from the evaluator's perspective<sup>17</sup>. Fuzzy logic plays a crucial role in analyzing imprecise and uncertain data. Fuzzy logic is founded on fuzzy set theory and contains infinite truth values.<sup>12</sup>

This study demonstrates an effective approach to managing spent sample utilization. Cookies were formulated using HS 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 developed cookies incorporated with HS. The HS powder has a floury texture, distinct flavor, and functional characteristics such as a rich source of minerals, dietary fibers, and bioactive substances. However, there has not been any study reported on using HS flour in the preparation of cookies. Fuzzy logic was employed for both the sensory evaluation and the 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 during March to April from Imphal, Manipur, India. The proximate composition (% wet basis) of the 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 ( $1.31 \pm 0.11$ ), total carbohydrate ( $6.91 \pm 1.89$ ). After the supercritical extraction process, the HS was collected from the SFE system and stored at  $-20^{\circ}\text{C}$  until further uses.

### 2.2. Preparation of Heiyai cookies

The cookie dough was prepared using the standard ratio of 3:2:1 *ratio* of flour to fat to sugar and the specific ingredients are listed in **Table 1**. In a separate bowl, the butter was beaten thoroughly using a beater and then all the ingredients were gradually added and mixed thoroughly. HS were 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 HS (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, then placed in a preheated baking oven (SM-502) at  $180^{\circ}\text{C}$  and baked for 30 min. After baking, the cookies cooled on the baking sheet for a few minutes before being transferred to an airtight container. For further analysis, the cookies were stored at room temperature in an airtight container after cooling for 30 min.

**Table 1:** Ingredients used for the preparation of cookies

Sample	Wheat flour(g)	HS (%)	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

### 2.3. Quality attributes of the prepared cookies



### 2.3.1. Proximate Composition

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The proximate composition of the prepared Heiyai cookies was determined using the AOAC (2020) method<sup>2</sup>. Precisely, the moisture content was measured utilising a hot air oven at 105°C. The ash content was estimated using a muffle furnace set at 550°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 TPC and Antioxidant activity

Heiyai cookie extract was obtained using 80% methanol as a solvent with slight modification according 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.

### 2.2.3. Total Phenolics Content (TPC)

The TPC of cookies methanolic extract was estimated using a method mentioned by Hussain et al.<sup>13</sup>. About 500 μL of each extract was taken, and 0.5 mL of Folin-Ciocalteu reagent was diluted (1:2) in distilled water, and 2 mL of 7% Na<sub>2</sub>CO<sub>3</sub> was added to the mixture, and incubated for 30 min. The 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)/ g of cookie extract.

### 2.2.4. DPPH Free radical scavenging activity assay

About 500 μL of Heiyai cookie extract was mixed with 3 mL of prepared DPPH solution in the test tube and incubated in the dark for 15 min. The absorbance was measured at 517 nm and 80% methanol was used as a blank sample<sup>11</sup>. The DPPH free radical scavenging activity was determined by the using below Eq. (1)

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



Where,  $A_{control}$  = Absorbance of the control sample and  $A_{sample}$  = Absorbance of the control sample. View Article Online  
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### 2.2.5. Mineral analysis

About 0.5g of Heiyai cookie powder was placed in a Kjeldahl tube, and a 20 mL solution of a 3:1 ratio of sulfuric acid ( $H_2SO_4$ ) and nitric acid ( $HNO_3$ ) was added to the tube. The digestion process was performed for approximately 4 h at  $420^\circ C$ , continuing 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  $0.2\mu m$ . Once the filtration was completed, the analysis was conducted using ICP-OES. The results are expressed in mg/kg.

### 2.2.6. Colour analysis

The colour values of the Heiyai cookies were analyzed by using a Colourimeter (Ultrascan VIS, Hunterlab, USA). The colourimeter was calibrated using a white plate. The colourimeter 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.

### 2.2.7 FTIR measurements

The Heiyai cookie powder samples were subjected to FTIR analysis using method as described by Singhal et al. (2024). The dried sample mixed with potassium bromate and hydraulic pressed to form pellet. The pellet was scanned FTIR spectrophotometer (Nicolet Instruments 410 FTIR, Thermo Scientific, USA) frequency range of  $400-4000\text{ cm}^{-1}$ . The measurement was done in transmittance mode.

### 2.2.8. Sensory evaluation

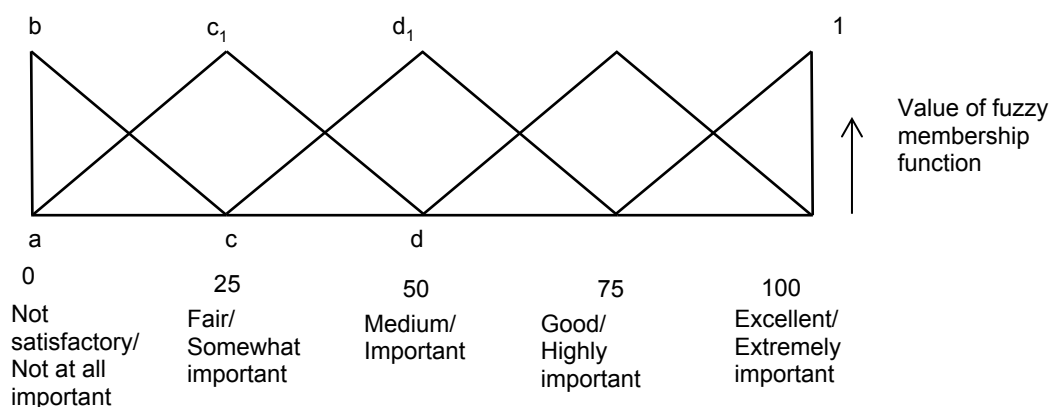
A total of 30 semi-trained panellists conducted the sensory evaluation of the Heiyai cookies. The panellists comprised of both male and female participants aged above 18 and not having any medical/health conditions that impact their sensory judgement skill. The sensory panellists were selected based on the interests and were initially trained about the different quality attributes of the cookies and different terms used in the sensory scale (ASTM STP-758, 1981)<sup>3</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

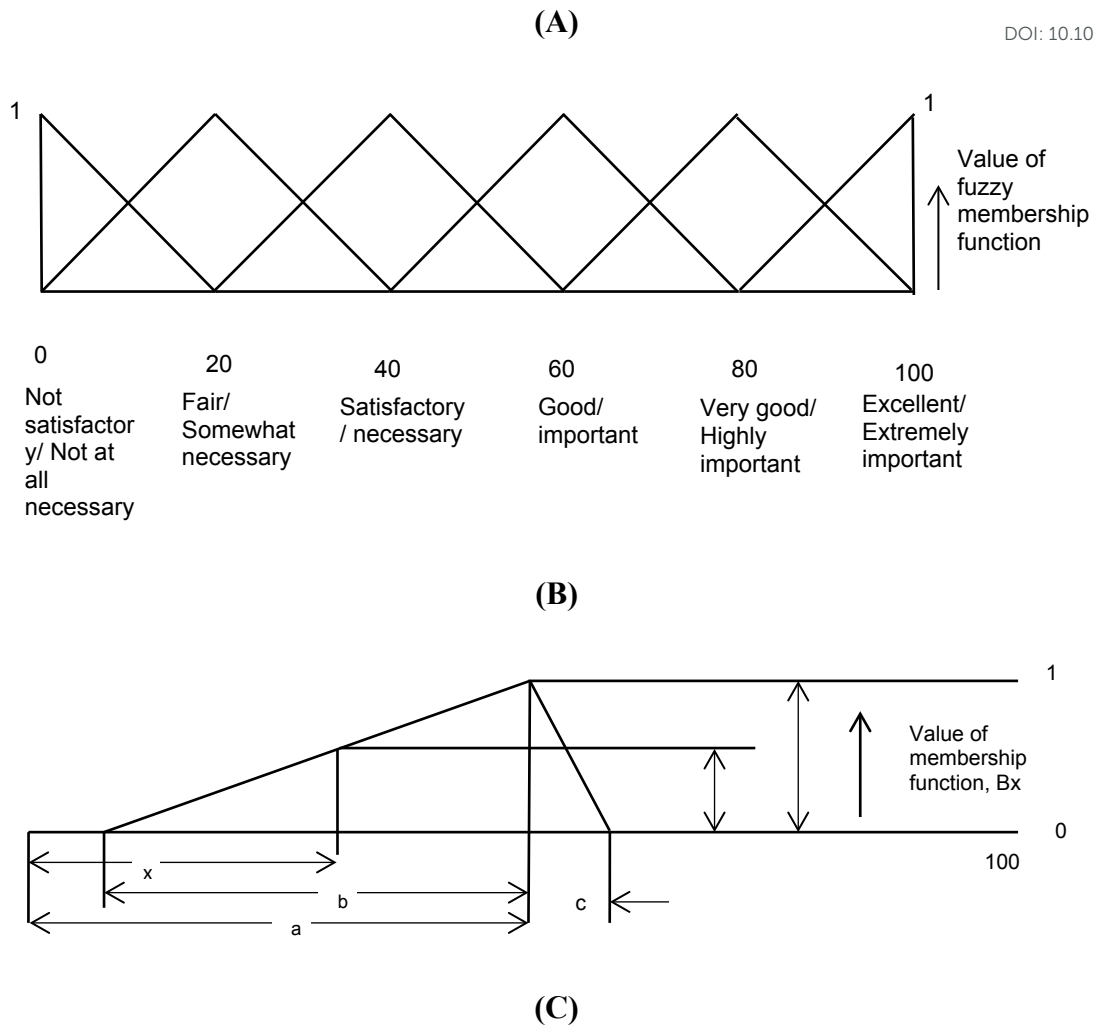


coded as HC1, HC2, HC3, HC4, and HC5. Panellists were instructed to rate the samples as 'poor', 'fair', 'medium', 'good', and 'excellent' for assessments of quality attributes and assign each quality attribute of cookies in general in terms of tick mark against the appropriate choices.

The sensory evaluation used a five-point language scale to gather responses. In Figure 1(A), the numerical values 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 described the scoring methodology to be used using a score chart from the sensory evaluation. 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 Figure 1(B).

The fuzzy modelling of sensory evaluation involves several key steps: (a) determining overall sensory scores for cookies samples using triplets, (b) membership functions calculation on a standard fuzzy scale, (c) calculating overall membership functions on a standard fuzzy scale for Heiyai cookies, (d) estimating similarity values and ranking the Heiyai cookies, (e) conducting a general ranking of quality attributes for Heiyai cookies, and the final step involves (f) ranking the quality attributes of individual Heiyai cookies <sup>22</sup>.





**Figure 1:** (A) Representation of triangular membership function distribution pattern of sensory scale (B) Standard fuzzy scale (C) Graphical view of one of the overall sensory scores as a triplet (a, b, c) (Adapted and modified from: Swami Hulle (2015)<sup>25</sup> and Das (2005)<sup>26</sup>)

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

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

Where  $HC_i$  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 represents the distance from the first number to the right. The second and third numbers members of the triplets had a membership function value of zero, while the first number had a membership function value of one<sup>24</sup>. The triplets ( $a b c$ ) for each triangular membership are displayed in Figure 1(C). and the quality attribute triplet values of the Heiyai cookies was determined by Equation (1) In general, corresponding ratio of each triplet to the maximum of the sum of the triplet sides was used to determine the triplet of relative weightage ( $W$ ) for the quality qualities. Using Equation (3), the overall sensory attribute ( $C_i$ ) for the sample of  $i^{\text{th}}$  Heiyai cookies was calculated.

$$OHC_i = \sum_{k=1}^6 (HC_i \text{ for } k^{\text{th}} \text{ attribute}) (W \text{ for } k^{\text{th}} \text{ attribute}) \quad (3)$$

Where  $k$  denotes six sensory attributes examined.

The triplet ( $a b c$ ) was multiplied by the delete triplet ( $o p q$ ) using Eq. (3), applying the triplet matrix multiplication rule.

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

For every standard fuzzy scale, a triangle distribution pattern was used to generate the membership function (F1 -F6) as shown in Eq. (5). The membership function has a maximum value of 1 and is composed of a set of ten numbers, shown in Figure 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  $F_6 = (0, 0, 0, 0, 0, 0, 0, 0, 0.5, 1) \quad (5)$

The value of membership function  $B_x$  for a given value of  $x$  on the abscissa can be expressed as follows: Equation 5.6. 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>20</sup>.

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



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

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$B_x = 0$  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 Eq. (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 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 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>19</sup>. Based on the size of  $S_m$  values, a similar process was used to rank the quality attributes of Heiyai cookies.

### 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, analysis of variance (ANOVA) and Duncan's multiple range tests were performed using IBM SPSS software.

## 3. Result and Discussion

### 3.1. Proximate composition of Heiyai cookies

The proximate composition of Heiyai cookies in five different formulations prepared using SFE HS 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 HS added to the cookies. A similar study by Ikuomola et al.<sup>14</sup>, the moisture level of cookie samples made



from blends of wheat flour and malted barley bran ranged from 3.34% to 4.6%. The study by Gernah et al.<sup>6</sup> revealed a similar pattern, with cookies prepared from wheat-brewer's spent grain flour blends having 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>1</sup>. Additionally, the low moisture level (less than 10%) ensures high storage stability by preventing microbial spoilage<sup>15</sup>. This is likely due to the hygroscopic nature of the residue powder, which absorbed moisture from the surroundings.

Similarly, the ash content of Heiyai cookies increased from 1.36% to 2.78%, while the control sample (HC1), prepared without HS, showed decreased. The increase in the ash content could be due to the mineral-rich HS 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>18</sup>. The result indicated that the substitution of HS at various levels varied significantly ( $p < 0.05$ ) in the fiber content of Heiyai. The sample HC5 had the highest fiber content (3.28%), whereas HC1 had the lowest (1.30%). The findings show that the fiber content of Heiyai cookies rises with a higher concentration of HS. The crude fiber content of the cookies varied from 1.36 to 3.39%. The control sample (HC1) contained the lowest amount of crude fiber, consisting only of refined wheat flour. This could be explained by the high crude fiber content of 5.9% found in Heiyai fruit<sup>16</sup>. In comparison, the other samples that included HS exhibited a higher concentration of crude fiber than the control sample (HC1).

**Table 2:** Proximate composition of Heiyai cookies

Sample	Moisture	Ash	Crude fat	Crude fiber	Protein	Carbohydrate
HC1	2.04 ± 0.05 <sup>c</sup>	1.36 ± 0.01 <sup>c</sup>	30.31 ± 0.12 <sup>d</sup>	1.30 ± 0.06 <sup>c</sup>	12.76 ± 0.20 <sup>b</sup>	50.47 ± 0.26 <sup>c</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>

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

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 HS substitution



exhibited lower fat amounts. When comparing all the samples containing HS, the fat contents of HC1 and HC5 were significantly different ( $p \leq 0.05$ ).

The amount of carbohydrate content of the cookies was generally high, ranging from 50.47% in sample HC1 to 59.11% in sample HC5. The cookies that contained the most HS 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 sample HC5, which had a greater addition of HS and less wheat flour, exhibited the lowest protein content. The substitution of HS led to a decrease in the protein content of the samples.

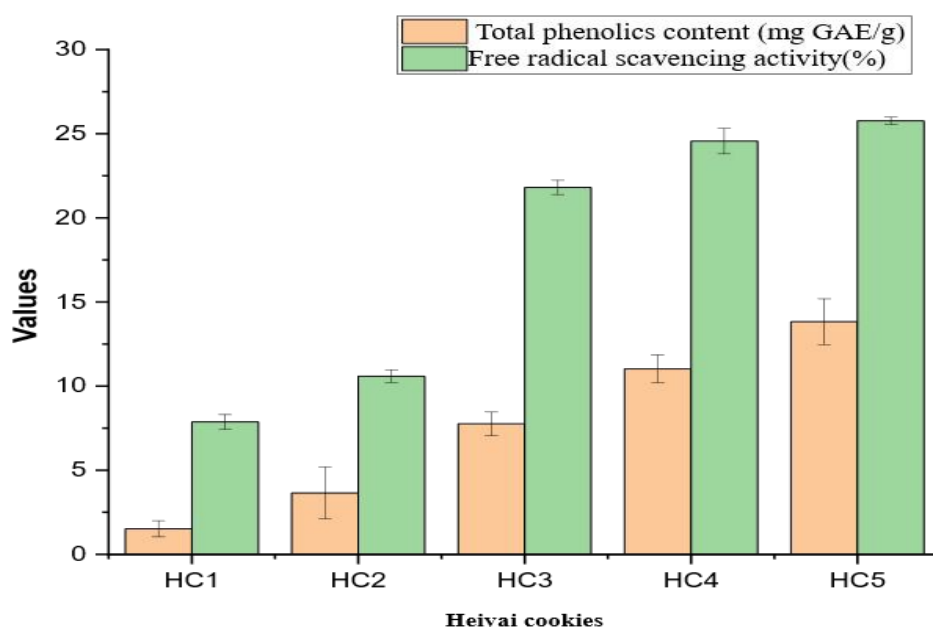
The presence of acrylamide was assessed using qualitative assessment using FTIR of sample containing highest and lowest concentration of Heiyai spent. The observed peaks around  $3236 \text{ cm}^{-1}$  correspond to N–H stretching vibrations, while peaks near  $2935 \text{ cm}^{-1}$  are attributed to 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 supplementary file (Figure S2). However, due to spectral overlap in complex heterogeneous matrices confirmatory chromatographic analysis is required for better result analysis and quantification<sup>29</sup>.

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

Total phenolic content (TPC) is a key indicator of antioxidant potential in functional food products. In this study, the effect of incorporating HS on the TPC of cookies was evaluated. The cookie sample with the highest concentration of HS (HC5) had the highest total phenolic content ( $13.83 \pm 1.36 \text{ mg GAE/g}$ ), while the control (HC1) had the lowest ( $1.52 \pm 0.48 \text{ mg GAE/g}$ ). A consistent pattern was found, with phenolic content increasing in proportion to the amount of HS incorporated (Figure 2). The phenolic compounds reported in *E. latifolia* are gallic acid, p-Coumeric, m-Coumeric, Ferulic acid, Chlorogenic acid, Caffeic acid, Catechin<sup>28</sup>.



The evaluation of free radical scavenging activity in cookies formulated with varying concentrations of HS was conducted and compared to the control sample. The DPPH radical scavenging activity was estimated as Şahin (2023)<sup>27</sup> reported better results for similar cookies incorporated with *Elagnus* fruit pulp and DPPH was found to be better indicator of antioxidant in their study. The results are presented in Figure 2. The free radical scavenging activity ranged from 7.87 to 25.77%. The findings indicated that replacing HS resulted in greater DPPH free radical scavenging activity than the control sample (HC1). Additionally, the cookie (HC5) with 15% HS exhibited the highest antioxidant activity. The result found that the DPPH free radical scavenging activity increased significantly with the increasing amount of all five levels of HS of the cookies.



**Figure 2:** Total phenolic content and free radical scavenging activity of the Heiyai cookies. All data are the mean  $\pm$ SD of three replicates.

Since Heiyai fruit is a significant source of total phenolic contents, adding it to cookies increased their DPPH free radical scavenging activity. Total phenolic contents and free radical scavenging activity are directly correlated. A similar pattern was found in the study conducted by Hussain et al.<sup>12</sup>. There are limited studies available on the use of *Elaeagnus latifolia* L. and similar species in products like cookies. Şahin (2023)<sup>27</sup> formulated oleaster (*Elaeagnus angustifolia* L.) parts in cookies and reported similar observations like improved phenolics content and antioxidant capacity. The higher antioxidant may be attributed the sample composition.

### 3.3. Colour analysis of Heiyai cookies



The colour parameters for five cookies formulated with different concentrations of HS, each containing 0%, 2%, 5%, 10%, and 15% of HS are shown in Table 3. The sample HC1 (0%) exhibited the highest  $L^*$  value among all the other formulations. As the amount of HS increased, the cookies' lightness ( $L^*$ ) decreased, and thus the HC5 had the lowest  $L^*$  value. This means that the cookies became darker as the level of SFE HS increased. The colour of the HS on themselves 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 4. On the other hand, the redness value and the yellowness 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 HS to the cookies containing the highest level of HS (HC5). Overall, the colour data indicated that the cookies appeared significantly darker due to an increase in HS. The photographs of cookies samples incorporated with HS are available in supplementary file.

**Table 3:** Colour analysis of Heiyai cookies

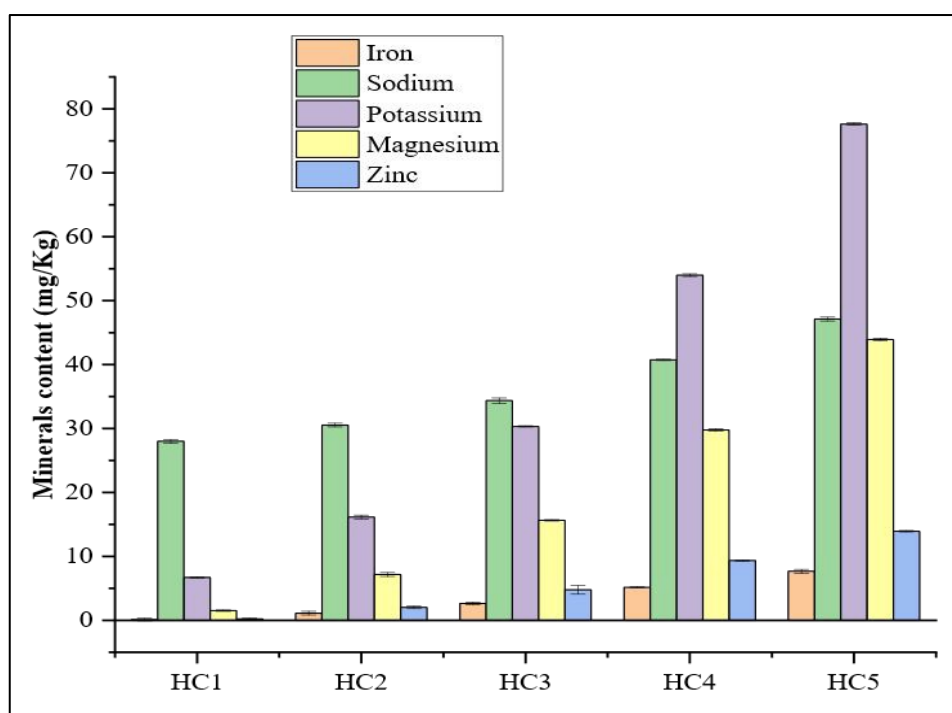
Sample	$L^*$	$a^*$	$b^*$
HC1	$73.72 \pm 1.42^a$	$2.367 \pm 0.54^c$	$26.12 \pm 1.62^d$
HC2	$69.31 \pm 0.92^b$	$8.27 \pm 0.42^d$	$29.50 \pm 0.60^c$
HC3	$66.17 \pm 0.08^c$	$12.67 \pm 0.31^c$	$32.59 \pm 0.57^b$
HC4	$61.65 \pm 0.22^d$	$16.25 \pm 0.07^a$	$54.77 \pm 0.24^a$
HC5	$48.28 \pm 0.12^e$	$14.06 \pm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

### 3.4. Mineral composition of Heiyai cookies

The mineral analysis was conducted on five different cookies made with varying levels of HS. The findings are presented in Figure 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 HS. There were substantial differences in the iron content among the cookie samples; HC5 had the highest concentration ( $0.684 \pm 0.03$  mg/kg), while HC2 had the lowest ( $0.102 \pm 0.67$  mg/kg). The sodium concentrations varied from  $23.79 \pm 0.12$  mg/kg (HC2) to  $28.71 \pm 0.03$  mg/kg (HC5). HC5 had the highest potassium levels at  $13.46 \pm 0.01$  mg/kg, while HC2 had the lowest at  $5.965 \pm 4.016$  mg/kg. Compared to the 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  $1.547 \pm 0.21$  mg/kg (HC4) to  $1.265 \pm 0.54$  mg/kg (HC2). HC4 had the highest magnesium level, even with the little variations. Both HC1 and HC5 showed consistent zinc levels at 0.208 mg/kg, while HC3 exhibited the lowest zinc concentration at  $0.150 \pm 0.65$  mg/kg. 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 HS may explain the differences in the mineral composition of the samples. The mineral content of the samples increased effectively as the concentration of HS increased. These results suggest that some samples have potential as functional foods with enhanced nutritional profiles.



**Figure 3:** Analysis of the mineral composition in Heiyai cookies. All data are presented as the mean  $\pm$  standard deviation (SD) of three replicates.

### 3.5. Sensory evaluation of Heiyai cookies by fuzzy logic

A total of 30 panelists assessed the sensory qualities of five different Heiyai cookies (HC1, HC2, HC3, HC4, and HC5), yielding significantly divergent evaluations. Sensory and quality attributes in general were utilised to calculate the triplet's correspondent with them based on the equation in (1). 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 Eq. (4) The triplet acquired was the overall sensory score for every sample is shown in Table 4. was

determined using Equation (3).

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

HC1					
Sensory scale	Colour	Flavour	Taste	Texture	Appearance
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
HC2					
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
HC3					
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
HC4					
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
HC5					
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



The values of the membership function were calculated using the equation presented in Equation 5.6. and the six-point sensory scale expressed as membership function values (F1, F2, F3, F4, F5, and F6) on a standard fuzzy scale Eq (5). Quality attributes and quality attributes in general for each Heiyai cookie sample were calculated and all values are shown in **Table 6**, respectively.

### 3.5.1. Ranking of Heiyai cookies according to the similarity values

The similarity values of the five different concentrations of Heiyai cookies developed using HS are shown in Table 5. The HC3 developed by incorporating 5% of HS showed the highest similarity value of 0.658, categorizing it as 'very good.' The cookie sample (HC2) also placed second rank with a similarity value of 0.649, falling into the 'very good' category and bearing significant similarity to HC2. The third preferred was HC1(control sample) obtained a similarity value of 0.680, placing it in the 'good' category. And both HC1 and HC5 were classified in the 'good' category. Lastly, the lowest similarity value was observed in HC4 at 0.789, positioning it in the 'fair' category. Heiyai cookies were ranked based on the highest similarity values for five samples. The results showed that HC3 (very good) > HC2(very good) > HC1 (good) > HC5(good) > HC4 (fair).

**Table 5:** Similarity values of cookies 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

### 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 in general for each of the Heiyai cookies

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The quality attributes of each 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, samples HC4 and HC5 were rated as being in the "satisfactory" category, with similarity values of 0.828 and 0.762, respectively.

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

In the HC4, the judges 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 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.

## 4. Conclusion

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



## 5. References

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**Data availability**

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

