



## Research Article

# Formulation of Nutraceutical Biscuits Based on Dried Spent Coffee Grounds

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## Abstract

**Background and Objective:** Spent coffee grounds (SCGs) are the most abundant residual material (45%) obtained during the instant coffee production. Therefore, around 6 million tons of SCGs generated per year. The purposes of the present study consisted in evaluating the use of SCGs as innovative functional food ingredient in bakery products and study the effect of SCGs and their chemical constituents on the sensory properties, volatile constituents and rheological properties of the final product. **Materials and Methods:** Effect of mixing wheat flour with 2, 4 and 6% SCGs on rheological properties of the obtained dough was evaluated, then chemical, texture profile and sensory properties of the final product were studied. **Results:** Rheological properties of mixed wheat flour with SCGs dough was affected slightly with the higher dietary fiber content. Moisture content of SCGs reached to 58.98%, therefore, was dried to reach 7.47% to control the microbial activity. Protein, ash, crude fiber and total carbohydrate of SCGs were 8.97, 2.77, 51.86 and 78.5%, respectively. Also, SCGs is good source of lipids (13.89%). Chemical composition of biscuit indicated that increasing mixing level of SCGs (2-6%) has shown good enhancement in fiber and ashes compared to control sample. Volatile sensory active compounds of biscuits and its supplemented mix with SCGs was evaluated. Thirty-eight volatile compounds was separated and identified in the biscuit samples by using SPME/GC-MS analysis. Hunter color parameter of produced biscuit indicated that whiteness ( $L^*$ ) of control sample reached to 71.17, while mixing SCGs with wheat flour at levels 2, 4 and 6% decreased  $L^*$  to 66.31, 54.88 and 43.88, respectively. Also, redness degree ( $a^*$ ) was lower in wheat flour (0.45) than SCGs (3.57). Therefore,  $a^*$  value of biscuit increased with increasing mixing level of SCGs. Sensory properties of biscuits showed that increasing SCGs level decreased significantly the color score but overall acceptability not affected significantly. **Conclusion:** Spent coffee grounds contains sufficient amounts from dietary insoluble fibers, protein, lipids, ashes and lower glycaemic sugars. Mixing wheat flour with SCGs affected slightly the rheological properties of dough and overall acceptability of the final product. Therefore, the obtained biscuit could be recommended to patients with obesity-related diseases and diabetes in addition to people looking for foods intake with reduced energy.

**Key words:** Spent coffee grounds, nutraceutical biscuits, rheological properties, volatile compounds, sensory properties

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**Data Availability:** All relevant data are within the paper and its supporting information files.

## **INTRODUCTION**

Coffee represented one of the most popular beverage in the world with annual consumption exceeds 400 billion cups, whereas more than 450 million cups of coffee are consumed in the US daily<sup>1</sup>. Coffee is cultivated in more than 50 developing countries in South and Central America, Africa, Asia and the Caribbean. The ICO production report for crop year 2016/17 showed 153.9 million bags (60 kg bag), with 97.3 million bags of Arabica type and 56.6 million bags of Robusta one<sup>2</sup>. Simply, coffee is a drink prepared by extraction the soluble material of roasted coffee grounds using boiling water. Different coffee brewing techniques applied in the world based on taste and consumer demands e.g., filtration-percolation technique<sup>2</sup>. Coffee industry as one of the most important agro-industrial and food sectors produce large quantities of waste, in both liquid and solid forms<sup>3</sup>. Spent coffee grounds (SCGs) are the most abundant residual material (45%) obtained during the treatment of coffee powder with steam or hot water for the instant coffee production<sup>4</sup>. Therefore, around 6 million tons of SCGs generated per year, since 50% of the global coffee production is used for soluble coffee preparation<sup>5</sup>.

From the nutritional point of view, SCGs have an important and concerned content of dietary fiber (47.3 g/100 g SCGs), composed mainly of insoluble fiber (88%)<sup>5</sup>. Polysaccharides constitute the other carbohydrate resource found in SCGs with (13.1%), while free glucose found in an insignificant concentration (0.04 g/100 g SCGs) which means a lower content of glycaemic sugars<sup>5</sup>. The ashes content of SCGs is (1.30%) including many elements e.g., potassium, sulfur, calcium, iron, magnesium, phosphorus, copper, manganese, boron and others<sup>6</sup>. Unsaturated fatty acids are the major lipid content of SCGs, which is ranged between 11-20%<sup>7</sup>. While due to the different conditions used during instant coffee preparation, protein content vary between 6.7-9.9<sup>8</sup> to 12.8-16.9%<sup>9</sup>.

Nowadays, under the huge political and social pressure, a lot of efforts have been made to reduce and avoid the pollution generating through industrial activities. However, in spite of the promising nutritional facts of SCGs, they are used in industrial fields as fuel in boilers due to its high calorific power<sup>10</sup> as an animal feed for pigs, ruminants, chickens and rabbits<sup>11</sup> and as a potential source for to generate fuel pellets and biodiesel<sup>12</sup>.

With respect to the modern lifestyles and consumers seek healthy foods, lower glycaemic index as well as lower caloric content of the food, became a main concerns

nowadays in addition to the dietary fiber content and their role in the balanced nutrition. Lower glycaemic foods are strongly recommended by the health officials who struggle for solutions to obesity epidemic and related diseases e.g., diabetes<sup>13</sup>. In this sense, the purposes of the present study consisted in evaluating the use of SCGs as innovative functional food ingredient in bakery products and study the effect of SCGs and their chemical constituents on the sensory properties, volatile constituents and rheological properties of the final product.

## **MATERIALS AND METHODS**

**Materials:** Wheat flour (72% extraction) was purchased from the North Cairo Flour Mills Company, Egypt. Spent coffee grounds (SCGs) were kindly supplied by Misr Cafe, 10th of Ramadan industrial city, Egypt, then stored at -20°C up to use. Shortening, skimmed milk, ground sugar, vanilla, eggs and baking powder were obtained from the local market of Cairo, Egypt.

**Preparation of flour and their blends:** The biscuits were prepared by mixing 100 g wheat flour (72% extraction) and their blends containing 2, 4 and 6% SCGs. Biscuit formula was as follows: 100 g flour, 40 g sucrose, 15 g shortening, 0.93 g salt, 1.11 g sodium bicarbonate and 1 g vanilla.

**Rheological properties:** Rheological properties of different dough blends were evaluated using farinograph according to AACC<sup>14</sup>. The viscoelastic properties of the prepared SCGs and wheat flour were examined using a Rapid Visco Analyser-4 (Newport Scientific, Australia) according to AACC<sup>14</sup>.

**Analytical methods:** Moisture, ash, fiber, protein and fat of raw materials and different biscuits were determined according to AOAC<sup>15</sup>. Total carbohydrates were calculated by difference.

**Baking quality of biscuits:** Volume (cm<sup>3</sup>) and weight (g) of three biscuits samples of each treatment were recorded. Specific volume was calculated by dividing of the volume to weight, diameter, height and Spread ratio (diameter/height) according to the method described in AACC<sup>14</sup>.

**Color determination:** Color parameters (L\*, a\* and b\*) of wheat flour, SCGs and their biscuit product at different mixing levels were determined using a spectro-colorimeter

(Tristimulus Colour Machine) with the CIE lab color scale (Hunter, Lab Scan XE-Reston VA, USA) in the reflection mode. The instrument was standardized with white tile of Hunter Lab Cooler Standard (LX No.16379):  $X = 72.26$ ,  $Y = 81.94$  and  $Z = 88.14$  ( $L^* = 92.46$ ,  $a^* = -0.86$ ,  $b^* = -0.16$ ) [29].

**Texture analysis:** Texture of the baked biscuit samples was performed texturometer, model Brookfield, CT3-10 kg, USA, equipped with a cylinder probe (TA.AACC36). Texture profile analysis (TPA) was conducted to determine hardness, adhesiveness, resilience, cohesiveness, springiness, gumminess and chewiness. The analyzer was set to perform two cycle measurements to give a two bite texture profile curve. Trigger load and test speed were 9.00 N g and 2.5 mm sec<sup>-1</sup>, respectively.

**Organoleptic evaluation of biscuits:** Organoleptic characteristics of biscuits were evaluated with some modifications, according to Hussein *et al.*<sup>16</sup> by 15 trained panelists. The tested characteristics were color (20), texture (20), flavour (20), taste (20), appearance (20) and overall acceptability (100).

**Headspace solid-phase microextraction (HS-SPME):** About 10 g of biscuits samples were introduced into a 20 mL SPME vial. The SPME device coated (fused-silica fiber) with a 100 µm layer of polydimethylsiloxane (Supelco, Bellefonte, PA, USA) was used for extraction of the volatiles and the vial was sealed with a silicone septum. The samples were exposed to the SPME vial at 60°C for 30 min, then immediately introduced in the gas chromatography injector. The above method was reported and optimized by Farouk *et al.*<sup>17</sup>.

**Gas chromatography-mass spectrometry analysis (GC-MS):** Separation of cakes aroma components was performed on Trace GC Ultra Chromatography (Thermo Scientific, USA), equipped with ISQ-Mass (Thermo Scientific, USA) and 60 m × 0.25 mm × 0.25 µm thickness TG-5MS capillary column (Thermo Scientific, USA). The column separation was programmed from 50°C with hold time 3 min and temperatures increase at rate 4°C min<sup>-1</sup> to 140°C with hold time 5 min, then at rate 6°C min<sup>-1</sup> to 260°C with 5 min. isothermal hold. The injector temperature was 180°C, ion source temperature was 200°C and the transition line temperature was 250°C. The carrier gas was helium with constant flow rate 1.0 mL min<sup>-1</sup>. The mass spectrometer had

a scan range from m/z 40-450. Ionization energy was set at 70 eV. The identification of compounds based on matching with the MS computer library (NIST library version 2005), compared with those of authentic compounds and published data and the relative percentage of the aroma constituents was calculated from GC peak areas (using GC Software). A linear retention was calculated for each compound using the retention times of a homologous series of C6-C26 n-alkanes<sup>18</sup>.

**Statistical analysis:** The obtained results were evaluated statistically using analysis of variance as reported by McClave and Benson<sup>19</sup>.

## RESULTS AND DISCUSSION

**Chemical composition of SCGs:** Upon collection, the moisture content of SCGs used in this study was 58.98%, therefore, it was important to subject SCGs for the immediate drying in order to control the microbial activity which may degrade the product and moisture which can hinder the oil extraction process. After vacuum drying, moisture content of SCGs was 7.47% (Table 1), which is in concordance with the technological and quality recommendations that advise to set the moisture content lower than 10%<sup>4</sup>. According to Ratti<sup>20</sup>, dehydration is the removal of moisture from food material in order to reduce the microbial activity and avoid its deterioration, i.e., dehydration is an efficient technique of food preservation.

Due to both hydrophilic nature of inorganic minerals and different brewing conditions applied during instant coffee process, ashes concentration reduced from 4.6 to 0.8-3.5%<sup>4,9</sup>. The content of ashes in the present study showed the same trend, whereas their concentration decreased to (2.77%), in agreement with Mussatto *et al.*<sup>21</sup>.

SCGs represented a good source of lipids (13.89%) as shown in Table 1, since both Robusta and Arabica beans have higher percentages of lipids, 11-16 and 14-20% but they are hardly extracted by hot water during instant coffee production, therefore, remain in SCGs. Lipids contributed to the sensory profile of coffee, whereas linoleic acid (40%), palmitic acid (30%), oleic acid (10%) and stearic acid (7%) constitute the principal components of these lipids<sup>22</sup>.

With respect to the extraction technique and the concentrations of non-extracted constituents, SCGs showed a significant concentration of protein 8.97% (Table 1). Arabica and Robusta both have a protein content range from 8.5-12%<sup>23</sup> which is in accordance to study findings.

Table 1: Chemical composition of raw materials and biscuits (on dry weight basis)

Constituents (%)	Raw materials		Biscuits			
	Wheat flour	SCGs*	Control	T1: Wheat	T2: Wheat	T3: Wheat
				flour+2% SCGs*	flour+4% SCGs*	flour+6% SCGs*
Moisture	11.90±0.10	7.47±0.32	25.76±0.19	26.30±0.22	27.02±0.29	27.88±0.32
Protein	12.05±0.25	8.97±0.11	8.23±0.15	7.32±0.13	6.05±0.17	6.78±0.10
Fat	1.12±0.03	13.89±0.05	16.10±0.09	16.10±0.07	16.17±0.10	16.37±0.12
Ash	0.70±0.06	2.77±0.01	1.06±0.03	1.10±0.05	1.13±0.03	1.16±0.04
Crude Fiber	0.45±0.04	51.86±0.22	0.90±0.01	1.22±0.02	1.65±0.05	1.71±0.07
Carbohydrate	85.68±0.65	78.50±0.54	73.71±0.65	74.26±0.71	75.00±0.62	73.98±0.79

Mean ± Standard deviation. \*Spent coffee grounds

Table 2: Effect of mixing wheat flour (WF) with SCGs on Hunter color parameters of biscuits

Samples	Raw materials		Biscuits form				LSD at 5%
	Wheat flour	SCGs	Control	WF with 2%SCGs	WF with 4%SCGs	WF with 6%SCGs	
L	92.33±1.11 <sup>a</sup>	20.05±0.04 <sup>f</sup>	71.17±2.15 <sup>b</sup>	66.31±1.22 <sup>c</sup>	54.88±2.18 <sup>d</sup>	43.88±1.95 <sup>e</sup>	4.253
A	0.45±0.02 <sup>e</sup>	3.57±0.05 <sup>b</sup>	2.53±0.04 <sup>d</sup>	2.92±0.05 <sup>a</sup>	3.41±0.06 <sup>b</sup>	5.62±0.03 <sup>a</sup>	0.095
B	9.53±0.13 <sup>c</sup>	6.58±0.06 <sup>f</sup>	24.52±0.91 <sup>a</sup>	21.17±1.20 <sup>b</sup>	18.12±1.07 <sup>c</sup>	15.49±0.87 <sup>d</sup>	1.895

Mean ± Standard deviation, Mean values in columns with different letters are significant at 0.05 probability level

In agreement with Vardon *et al.*<sup>24</sup>, dietary fibre was found as the main constituent among SCGs components with 51.86% and the abundant in carbohydrate fraction 78.5% (Table 1). Insoluble fiber was represent the main component of SCGs dietary fiber (88%) which is predictable with respect to brewing processing and extract the majority of soluble fiber during the production of instant coffee<sup>5</sup>. The SCGs are rich in lignocellulose, a covalently bonded network of lignin, cellulose and hemicellulose polysaccharides that gives structural stability to the plant cell wall<sup>6</sup>. Dietary fiber is indigestible in human small intestinal but digested completely or partially fermented in the large intestine and therefore control on many of its functions with an important physiological effects on glucose, lipid metabolism and mineral bioavailability. Nowadays, dietary fibers are known to have a protective effect against many gastrointestinal diseases e.g., colon cancer, duodenal ulcer, cardiovascular diseases, stroke, diabetes, constipation, hemorrhoids, gastroesophageal reflux disease, diverticulitis, obesity and hypertension. Dietary fibers also have functional and technological properties that can be used in the formulation of foods due to their low energy values as well as numerous beneficial effects on human health<sup>25</sup>.

**Chemical composition of innovative biscuits:** Chemical composition of control and supplemented samples are presented also in Table 1. SCGs possessed a concerned quantity of fibre (51.86%) along with ashes (2.77%) which are a source for potassium, magnesium, calcium, sulfur and phosphorus<sup>6</sup>. Increasing addition of SCGs (2, 4 and 6%) has shown good enhancement in fibre and ashes in biscuits when compared to control (Table 1). Such increments revealed the

thermal stability of SCGs during food processing, which make them safe and suitable to be incorporated into food products. Martinez-Saez *et al.*<sup>5</sup> produced biscuits supplemented with many functional ingredients e.g., maltitol, oligofructose, stevia and SCGs. However, they were focused more on digestibility, safety and sensory properties for the innovated biscuits without a clear complete data concerning the chemical composition of the final product. Protein content was lower for supplemented samples in comparison to the control one with respect to its percentage on the raw materials, while fat content did not represent significant differences ( $p > 0.05$ ).

**Color attributes and evaluation of browning degree of innovative biscuits:** Most sensory active compounds generated during baking are products of nonenzymatic browning or Maillard reactions, so, the degree of browning was investigated both by instrumental and sensory techniques but from different aspects. Sensory assessors were not asked to give their opinions on the colour hue, however, the instrumental data were expressed as trichromatic parameters related to the colour hue. The color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) of raw materials and biscuit samples were evaluated and presented in Table 2. Scale range of whiteness ( $L^*$ ) is from 0 (black) to 100 (white),  $a^*$  scale extends from a negative value (green hue) to a positive value (red hue) and  $b^*$  scale from negative value (blue) to positive value (yellow).

Wheat flour characterized with its higher whiteness ( $L^* = 92.33$ ) as shown in Table 2, while SCGs was higher in darkness ( $L = 20.05$ ). Producing biscuit led to reach  $L^*$  value to 71.17 in control sample, while mixing SCGs with wheat flour at levels 2, 4 and 6% decreased  $L^*$  to 66.31, 54.88 and 43.88,

respectively. The decrease of lightness ( $L^*$ ) with increasing the concentration of the dark SCGs, was attributed to the less amount of light scattering. The degree of  $a^*$  was lower in wheat flour (0.45) than SCGs (3.57). Therefore,  $a^*$  value of biscuit increased with increasing mixing level of SCGs, where  $a^*$  value was 2.53 in control sample then increased significantly at mixing level 2, 4 and 6% to 2.92, 3.41 and 5.62. In contrary with  $a^*$  value,  $b^*$  value was higher in wheat flour (9.53) than SCGs (6.58), therefore, mixing wheat flour with SCGs at 2, 4 and 6% decreased significantly  $b^*$  value in biscuit from 24.52 in control sample to 21.17, 18.12 and 15.49, respectively. It is well known that, the increase of  $a^*$  and  $b^*$  corresponds to the increase in redness and yellowness, respectively, which occurs in the beginning of nonenzymatic browning or Maillard reaction<sup>26</sup>. However, the confounded results presented in Table 2, increase of  $a^*$  and decrease  $b^*$  values might be due to the brown color being too dark to be detected<sup>27</sup>. Therefore, such values cannot be taken as indicators for the development of browning reaction during our study. Sherwin and Labuza<sup>28</sup> reported that, the change of  $b^*$  value showed an increase over time and correlates well to brown pigment production, however, it is not the case during color development in fructose-glycine in glycerol model system at moisture level of 0, 5 and 10%. Again, Martinez-Saez *et al.*<sup>5</sup> reported a higher formation of Amadori compounds in commercial biscuits in comparison to the recipes supplemented with SCGs and other ingredients, suggesting faster rate of Maillard reaction early stage. This could be explained by the lower content of glycaemic sugars may found in supplemented recipes, which affected negatively on browning development.

**Volatile sensory active compounds:** Thirty-eight volatile compounds could be separated and identified in the biscuit samples by using SPME/GC-MS analysis (Table 3). These compounds were related to different chemical classes mainly aliphatic aldehydes (12), short chain and fatty acids (6), furans and furanones (7) and pyrazines (4). These findings are in agreement with Pasqualone *et al.*<sup>29</sup>, whom pointed to the origin of these volatiles from Maillard reaction, lipid oxidation or fermentation.

As 2/3-Methylbutanal and phenylacetaldehyde which were identified in the present study (Table 3) are known as strecker aldehydes derived from isoleucine, leucine and phenylalanine, respectively<sup>30</sup>. The increase in SCGs revealed a slight decrease in the strecker aldehydes concentrations, due to the lower reducing sugar content of SCGs which affected negatively the development of the early stage of Maillard reaction which lead to the formation of such aldehydes.

Lower glycaemic sugars could be easily degraded during nonenzymatic browning lead to the formation of many compounds e.g., 2,3-Butanedione and 2,3-Pentandione<sup>31</sup> (Table 3). However, the decrease in the amounts of such diketones upon supplemented biscuits with SCGs, revealed the lower glycaemic index of these residues.

The furans comprised the major class among the volatiles of both control and supplemented samples, whereas 2-Furfural constituted the predominant compound, with a significant decrease ( $p < 0.05$ ) on increasing SCGs level (Table 3). Furfural is a pungent and sweet but does not have a cookie-like, caramel aroma. However, 2-Ethyl-5-methylfuran and 2-Pentylfuran are lipid derived products with a non significant increase ( $p > 0.05$ ) on increasing SCGs. 2,5-Dimethyl-4-hydroxy-3(2H)-furanone is the only furanone identified on this study with caramel-like, sweet, fruity, nutty and burnt notes<sup>32</sup>.

Pyrazines are characterized with their roasted, nutty flavour properties<sup>33</sup>. Reaction of amino acids with sugar is the main route to the formation of alkyl pyrazines<sup>34</sup> but the inhibition or lower the browning rate due to lack of reducing sugars, may decrease pyrazines as seen in Table 3 with increasing SCGs.

As 2,3-Dihydro-3,5-dihydroxy-6-methyl-4(H)-pyran-4-one and its thermal dehydrated product, 3-Hydroxy-2-methyl-4(H)-pyran-4-one (maltol) have sweet-caramel note and well-known pyranones in cookies<sup>35</sup>. Addition of protein source to cookie dough promotes the generation of pyranones with a pleasant sweet aroma. Therefore, biscuits samples supplemented with SCGs showed a lower content of pyranones in comparison to the control sample (Table 3) due to the lower protein content of the supplemented recipes.

Oxidation of oleic acid, linoleic acid and linolenic acid, which are found in wheat flour and SCGs, is responsible for the formation of lipid degradation products found in the biscuit e.g., hexanal, nonanal, heptenal and 2,4-Decadienal<sup>33,36</sup>. The apparent increase in lipid-derived volatiles upon supplemented with SCGs may be attributed to the decrease in the formation of Maillard-derived products (Table 3).

**Sensory properties of biscuits:** The mean sensory scores of control and mixed biscuits with SGCs are shown in Table 4 and illustrated in Fig. 1. Significant differences ( $p < 0.05$ ) in color were observed between control and supplemented samples, whereas increasing of SCGs decreased significantly the color score due to their dark nature.

In spite of the differences recorded among samples in the identified volatiles which affected the aroma and taste,

Table 3: Volatile constituents of biscuits and its supplemented mix with SCGs

Compounds	RI*	Area (%)			
		Control	T <sub>1</sub>	T <sub>2</sub>	T <sub>3</sub>
2,3-Butanedione	619	7.18	5.64	4.88	4.18
2-Methyl butanal	641	2.18	1.47	1.12	1.04
3-Methyl butanal	650	5.54	4.18	3.88	2.99
Acetic acid	660	3.73	2.98	3.01	3.00
2-Pentenal	688	0.77	0.82	1.22	1.57
2,3-Pentanedione	702	0.91	0.42	0.38	0.21
Pyrazine	722	0.80	0.51	0.44	0.20
1H-pyrrole	758	0.13	0.08	0.09	0.07
2-Ethyl-5-methylfuran	766	0.41	0.50	0.49	0.47
Hexanal	779	4.52	6.61	7.17	8.01
3-Hexenal	813	0.23	1.33	2.04	3.08
Butyric acid	818	0.54	0.49	0.61	0.77
2-Methyl Pyrazine	828	2.55	1.68	1.31	0.62
2-Furfural	852	26.64	23.01	22.41	19.88
2-Hexenal	858	0.61	1.87	2.54	3.47
2-Furfuryl alcohol	863	2.64	1.99	1.01	0.54
4-Heptenal	900	4.08	5.75	6.08	6.87
Pyrazine, 2,5-dimethyl-	905	1.01	0.81	0.65	0.33
Pyrazine, 2,6-dimethyl-	913	0.95	0.72	0.42	0.21
2-Furfural, 5-methyl-	966	2.44	2.01	1.41	0.99
Hexanoic acid	970	0.14	0.25	0.48	0.71
2-Pentylfuran	993	2.41	2.48	2.62	2.01
Octanal	1005	2.77	3.18	4.09	5.11
2,4-Heptadienal	1011	0.92	1.11	1.88	2.14
Phenylacetaldehyde	1038	3.88	2.77	2.33	1.77
Phenylethanol	1060	3.24	2.05	1.88	1.06
2,5-Dimethyl-4-hydroxy-3(2H)-furanone	1077	1.21	0.68	0.35	0.11
Nonanal	1107	4.83	6.15	7.08	6.97
3-Hydroxy-2-methyl-4(H)-pyran-4-one	1111	2.01	1.12	0.99	0.44
2,3-Dihydro-3,5-dihydroxy-6-methyl-4(4H)-pyranone	1161	0.87	0.55	0.41	0.18
Decanal	1204	0.09	0.19	0.62	1.11
5-Hydroxy-2-methylfurfural	1251	0.12	0.07	0.08	0.04
γ-Butyrolactone	1300	1.12	0.68	0.33	0.18
2,4-Decadienal	1324	2.98	4.76	5.18	5.99
Decanoic acid	1376	0.77	1.23	2.07	3.11
Δ-Decalactone	1501	1.04	0.47	0.22	0.14
Tetradecanoic acid	1754	0.31	1.57	2.99	3.44
Hexadecanoic acid	1984	0.42	2.13	3.18	4.88
Total		96.99	94.31	97.94	96.19

\*RI: Retention index

however, no significant differences ( $p > 0.05$ ) could be found between the control and supplemented samples (2 and 4% SCGs). While mixing biscuits with up to 6% SCGs decreased significantly ( $p < 0.05$ ) aroma and taste attributes. Obviously, the odor and taste acceptability were affected by the presence of more glycaemic reduced sugars, cause of their responsibility for developing the browning reaction and the generation of compounds with sensory attributes e.g., roasted, sweet and caramel<sup>14,26</sup>.

Surprisingly, the texture of supplemented samples seems to be nearly as the control or better than it without significant differences ( $p > 0.05$ ), which may attribute to the higher emulsifying activity and stability of SCGs reported previously

by Ballesteros *et al.*<sup>6</sup>. The low appearance attribute for the biscuit sample mixed with 6% SCGs may be due to the darkening of the product. However, the overall acceptability showed no significant differences ( $p > 0.05$ ) among the examined samples (Table 4) which makes SCGs a promising functional ingredient with a great potential to be used in biscuit products.

**Rheological properties of dough:** Rheological properties of wheat flour and its mixtures with SCGs were evaluated using a Farinograph as shown in Table 5. The water absorption, arrival time, dough development time, dough stability and weakening of dough increased with an increasing level of

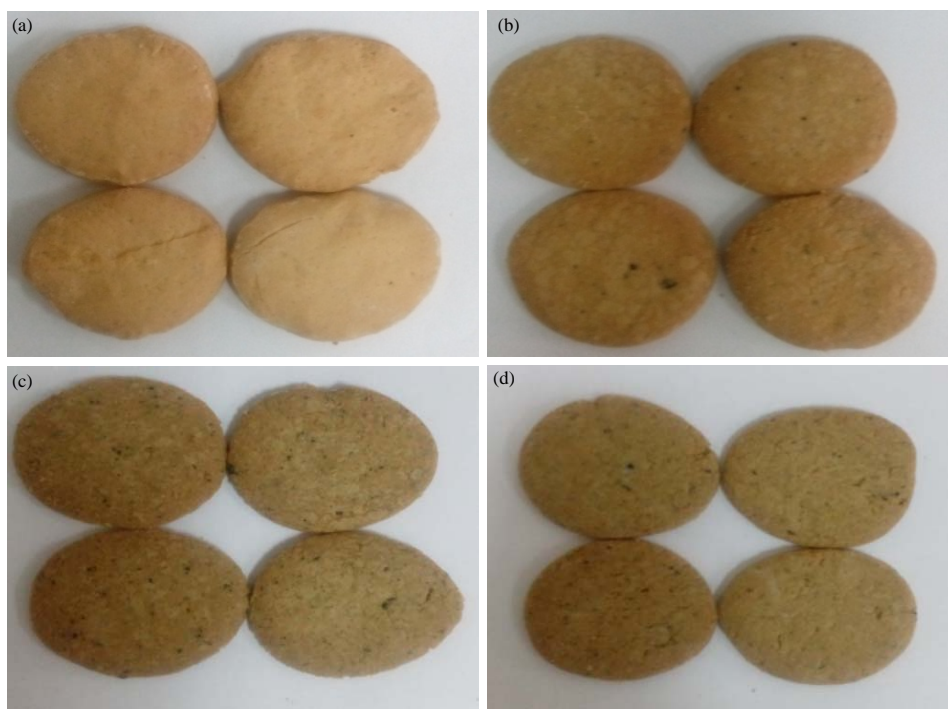


Fig. 1(a-d): Biscuits produced from wheat flour and its supplement with SCGs, (a) Control, (b) Biscuit mixed with 2% SCGs, (c) Biscuit mixed with 4% SCGs and (d) Biscuit mixed with 6% SCGs

Table 4: Effect of mixing biscuit with SCGs on its sensory properties

Sensory properties	Biscuit (control)	Biscuit mixed with SCGs			LSD at 5%
		2%	4%	6%	
Color (20)	18.15±0.51 <sup>a</sup>	16.35±0.63 <sup>b</sup>	15.01±0.91 <sup>c</sup>	13.20±0.86 <sup>c</sup>	1.154
Taste (20)	18.00±1.10 <sup>a</sup>	18.25±0.91 <sup>a</sup>	18.65±0.87 <sup>a</sup>	16.02±0.98 <sup>b</sup>	1.141
Odor (20)	18.21±0.61 <sup>a</sup>	18.05±0.96 <sup>a</sup>	17.82±0.74 <sup>a</sup>	16.74±0.85 <sup>b</sup>	1.340
Texture (20)	18.25±0.32 <sup>a</sup>	18.10±1.05 <sup>a</sup>	18.44±0.94 <sup>a</sup>	18.70±0.99 <sup>a</sup>	1.319
Appearance (20)	18.72±0.17 <sup>a</sup>	18.47±1.19 <sup>a</sup>	17.20±1.22 <sup>a</sup>	16.14±0.95 <sup>b</sup>	1.445
Overall acceptability (100)	90.09±3.66 <sup>a</sup>	88.11±3.42 <sup>a</sup>	87.62±2.98 <sup>a</sup>	87.14±3.71 <sup>a</sup>	4.689

Mean values in columns with different letters are significant at 0.05 probability level

Table 5: Effect of mixing SCGs powder with wheat flour on rheological properties of Farinograph

Rheological properties	Wheat flour	Wheat flour mixed with SCGs		
		2%	4%	6%
Water absorption (%)	62.0	65.0	67.0	69.0
Arrival time (min)	1.5	2.0	2.0	2.5
Dough development time (min)	2.0	2.5	2.5	3.0
Dough stability (min)	8.0	6.0	5.0	5.0
Mixing tolerance index (BU)	25.0	30.0	40.0	45.0
Dough weakening (BU)	20.0	60.0	80.0	80.0

SCGs added. This increment may be due to high fiber content of SCGs compared to wheat flour, where fibers tend to bind more water. Interacting of SCGs fibers with wheat flour constituents and the added water may caused a decreased in stability of dough as observed in Table 5. In this context, Kim *et al.*<sup>37</sup> reported that water absorption of dough increased as rice grain dietary fibers increased in the recipe.

Urooj *et al.*<sup>38</sup> and Hussein *et al.*<sup>39</sup> reported that increasing proportion of barley flour in the blend with white flour caused progressive increase in water absorption, arrival time and dough stability, with the addition of gelatinized corn flour to wheat flour. Whereas mixing tolerance index and dough weakening were increased by increasing the level of SCGs.

Table 6: Effect of mixing wheat flour with SCGs on baking quality of biscuits

Baking properties	Biscuit (control)	Biscuit mixed with SCGs			LSD at 5%
		2%	4%	6%	
Weight (g)	11.50±0.31 <sup>b</sup>	12.08±0.23 <sup>a</sup>	11.70±0.28 <sup>b</sup>	11.30±0.17 <sup>b</sup>	0.425
Volume (cm <sup>3</sup> )	18.50±1.00 <sup>b</sup>	21.00±0.50 <sup>ab</sup>	21.50±1.50 <sup>a</sup>	22.00±0.50 <sup>a</sup>	1.054
Specific volume (g cm <sup>-3</sup> )	1.61±0.08 <sup>c</sup>	1.76±0.08 <sup>b</sup>	1.84±0.05 <sup>b</sup>	1.95±0.09 <sup>a</sup>	0.122
Diameter (cm)	6.50±0.07 <sup>a</sup>	6.20±0.15 <sup>b</sup>	5.80±0.11 <sup>c</sup>	5.50±0.13 <sup>d</sup>	0.213
Height (cm)	1.00±0.03 <sup>d</sup>	1.20±0.05 <sup>c</sup>	1.30±0.07 <sup>b</sup>	1.50±0.09 <sup>a</sup>	0.012
Spread ratio (diameter/height)	6.50±0.05 <sup>a</sup>	5.17±0.07 <sup>b</sup>	4.46±0.03 <sup>c</sup>	3.67±0.01 <sup>d</sup>	0.651

Mean values in columns with different letters are significant at 0.05 probability level

Table 7: Effect of mixing SCGs with wheat flour on rheological properties of rapid visco-analyzer

Rheological properties of rapid visco-analyzer	Wheat flour	Wheat flour mixed with SCGs		
		2%	4%	6%
Peak1	2550	2016	1490	1350
Trough 1	1510	1100	880	730
Breakdown (cP)	1060	840	660	620
Final viscosity (cP)	2900	2415	1810	1550
Setback (cP)	1420	1210	1000	890
Peak time (min)	6.00	5.80	5.70	5.50
Pasting temperature (°C)	68.60	80.20	85.10	89.00

cP: Centipoise

Table 8: Effect of mixing biscuits with SCGs on its texture profile properties

Texture profile parameters	Biscuit (control)	Biscuit mixed with SCGs		
		2%	4%	6%
Hardness (n)	35.58	15.62	15.07	15.10
Adhesiveness (mJ)	7.70	0.70	0.40	0.30
Resilience	0.01	0.01	0.01	0.21
Cohesiveness	0.04	0.08	0.07	0.32
Springiness (mm)	0.00	0.01	0.00	0.05
Gumminess (n)	1.24	1.12	1.06	4.59
Chewiness (mJ)	0.00	0.00	0.00	0.05

**Baking quality of biscuits:** The effect of mixing wheat flour with SCGs on the baking quality of biscuits is shown in Table 6. Biscuit height is significantly increased with increasing mixing level of SCGs ( $p < 0.05$ ), while the volume showed a significant rise upon addition of SCGs generally. This effect may be due to the higher fiber content in SCGs as well as the emulsifying properties reported by Ballesteros *et al.*<sup>6</sup> and El-Shebini *et al.*<sup>40</sup>. Meanwhile, diameter and spread ratio were decreased significantly compared to the control as affected with SCGs.

**Effect of mixing wheat flour with SCGs on dough properties using rapid visco-analyzer (RVA):** Rapid visco-analyzer peaks of wheat flour and their blends with SCGs at different levels (2, 4 and 6%), in addition to their pasting properties are summarized in Table 7. The obtained data showed that, mixing wheat flour with SCGs led to decrease peak viscosity, trough (cP), breakdown (cP), setback (cP), final viscosity (cP) and peak time (min) while pasting temperature (°C) increased. It is well known that peak viscosity indicates the water holding capacity of starch and refers to the maximum viscosity

reached during the heating and holding cycle. It can be affected by the molecular structure of amylopectin<sup>41</sup>, starch, water concentration, lipids, residual proteins<sup>42</sup>, granule size<sup>43</sup> and instrument operating conditions<sup>44</sup>, pasting properties of starch are also affected by amylose and lipid contents and by branch chain-length distribution of amylopectin. Amylopectin contributes to swelling of starch granules and pasting, whereas amylose and lipids inhibit the swelling<sup>45,46</sup>. Furthermore, the amylopectin chain-length and amylose molecular size produce synergistic effects on the viscosity of starch pastes<sup>47</sup>.

**Texture profile analysis (TPA):** The texture profile (force vs. time) curve for the treatments was used for the estimation of hardness and resilience. Hardness is the peak force measured during the first compression cycle (i.e., first bite). Resilience is how well a product "fights to regain its original position". Resilience is measured as the ratio of area, covered under maximum force to base line and area, covered under initial point to the maximum force (Table 8). Measurement of biscuit texture in the texture analyzer showed that the hardness value



decreased when SCGs powder content in the biscuit formulation was increased. Resilience was also significantly reduced. According to Annor *et al.*<sup>48</sup>, the harder texture of the cookies is attributed to the increased protein content and its interaction during dough development and baking. Variation in texture profile analysis (TPA) due to various levels of SCGs powder incorporation might be due to lipid content as well as protein and starch quality. Sudha *et al.*<sup>49</sup> reported that fat coats the surface of the flour particles inhibiting the development of the gluten proteins. The free fat, therefore, disrupts the gluten network resulting in softer doughs<sup>50</sup>.

### CONCLUSION

Spent coffee grounds is the main byproduct of instant coffee process with concerned contents of dietary insoluble fibers, protein, lipids, ashes and lower glycaemic sugars. Mixing wheat flour with SCGs affected slightly the rheological properties of dough and overall acceptability of the final product up to 6% SCGs. Therefore, biscuit of 6% SCGs is more beneficial and could be recommended to patients with obesity related diseases and diabetes in addition to people looking for foods intake with reduced energy.

### SIGNIFICANCE STATEMENT

This study discovers the possibility of using SCGs as a functional ingredient in biscuit that can be beneficial for patients with obesity-related diseases and diabetes in addition to people looking for foods intake with reduced energy. Moreover, this study help the researcher to uncover the critical areas of SCGs waste that many researchers were not able to explore its beneficial in bakery products.

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