Ultrasonics Sonochemistry 109 (2024) 107015

Contents lists available at ScienceDirect

Ultrasonics Sonochemistry

journal homepage: www.elsevier.com/locate/ultson

# Valorizing date seeds through ultrasonication to enhance quality attributes of dough and biscuit, Part-1: Effects on dough rheology and physical properties of biscuits

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ARTICLE INFO

*Keywords:*
Date by-products
Ultrasound-assisted extraction
Rheological behavior
Biscuit quality characteristics
Texture
Microstructure

ABSTRACT

In the present study, non-conventional and green technology (ultrasonication) was utilized to recover bioactive compounds from the small, medium and large sized defatted date seed powder (DDSP) particles. Bioactive compounds recovered from DDSP and the remaining fiber-rich residue were incorporated as functional ingredient in the biscuit dough to enhance the functionality and the quality characteristics of the dough and biscuit. The polyphenolic extract and 2.5 %, 5 % and 7.5 % substitution levels of fiber-rich extraction residue were incorporated in formulations followed by investigating the effect on rheological, physical and microstructural properties of dough and biscuit. Loss and storage moduli, G'' and G', respectively, of dough increased with decreasing particle size and increasing substitution level while tan δ decreased with increasing substitution level of fiber-rich extraction residue. The smallest particles at 7.5 % substitution level resulted in the lowest creep strain value in dough. Hardness of the dough and biscuit increased with decreasing particle size and increasing substitution level of the residue. The 7.5 % substitution level of the smallest particle size resulted in the darkest dough and biscuit. Spread ratio and diameter of the biscuit decreased with increasing substitution level of the residue. The smallest diameter of 50.61 mm and spread ratio of 8.36 was observed in the biscuits substituted with the largest particle size with 7.5 % substitution level. Microstructural images of dough and biscuit revealed that the continuity of the gluten network was disrupted by the incorporation of the fiber-rich extraction residue. This study provided valuable insights into extracting bioactive components from date by-products using green ultrasonication technique and utilizing such compounds to improve functional attributes of bakery products, as a sustainable approach for valorizing date by-products.

## 1. Introduction

Bakery products are considered part of the daily food consumption category primarily because they are readily available and organoleptically acceptable. Among all the bakery products, biscuits play a vital role in daily diet of all age groups. Current population tends to go for more healthier diets because of the increasing disease rates due to lack of nutrients [1]. On this respect consumer demand for value-added food is increasing day by day. Hence, formulation of value-added biscuits could be a potential approach towards a healthier lifestyle in all age groups. There are several studies focused on improving the nutritional value of biscuits by incorporating plant materials due to the high nutrient profile of plants particularly fruits [2–4].

The global demand for utilizing food industrial by-products as food ingredients has increased significantly in the process of sustainable food product utilization. Most of the by-products of fruit processing industry exhibit good nutritional and bioactive compound profiles [5–7]. In line with the above-mentioned context, date by-products have shown potential to be utilized in value-added foods to improve their nutritional and functional properties. Date industry generates a vast amount of by-products including low quality dates, seeds, leaves, pomace and press cake. Among them, date seeds have shown a high nutritive value attributed by the rich profile of phytochemicals and dietary fibre [8–10]. Incorporating date seed powder (DSP) and/or polyphenolic extracts

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https://doi.org/10.1016/j.ultsonch.2024.107015
Received 28 May 2024; Received in revised form 2 August 2024; Accepted 3 August 2024
Available online 4 August 2024

have demonstrated significant outcomes in enhancing the nutritional value of food, specifically in bakery products [11–14].

To recover valuable compounds in a sustainable and green approach, ultrasound technology is considered to be an efficient food processing method which is recognized as environmental friendly, safe and non-toxic [15]. Improved efficiency, less time and solvent consumption are the major benefits of using ultrasound assisted extraction (UAE). Microbubbles are formed within the solvent through ultrasonic waves, resulting acoustic cavitation which helps in rupturing the cell walls of the matrix, enabling increased mass transfers to occur and enhancing extraction efficiency [16]. UAE has exhibited high efficiencies in extracting bioactive compounds including polyphenolic compounds [17,18]. Additionally, ultrasound waves can alter the structure and physical properties of food powders [19,20] thus enhancing their functionality and their application prospectus in different food systems. Extraction residue obtained after UAE have shown improved functional and physical properties [17]. Therefore, bioactive compound rich extracts obtained using UAE from DSP and the remaining fiber-rich extraction residue have higher potential to alter the functional properties and improve the nutritional content of bakery items compared to direct substitution of DSP.

Incorporation of external material apart from the usual ingredients of biscuits can impact either positively or negatively in the dough as well as the final product [2–4,21]. If used in the form of a powder, the particle size could affect on the gluten network of the dough [22]. Besides, the functional groups of the compounds that are added could interact with the compounds of the dough, resulting in changed behavioural patterns of the dough. The afore-mentioned effect is evident in biscuits fortified with fiber-rich sources [3,23–25]. Additionally, the colour attributes may vary which will have a drastic impact on the final product. Changes in physical properties in biscuits with the incorporation of DSP have been studied where the researchers observed significant changes in physical properties [26–28]. However, the effect on physical aspects of dough as well as biscuit, by incorporating ultrasonic-based dates seed polyphenolic extract and remaining fiber-rich residue of defatted date seed powder (DDSP) has not yet been reported. This approach covers the complete recycling and utilization of date seeds while employing sustainable and green processing techniques.

As such, the objective of this study is to examine the effects of incorporation of ultrasonic extracted polyphenols and remaining fiber-rich residue into biscuit dough and how it would affect the physical characteristics of the final product. The study explored the impact of varying particle sizes of DDSP in conjunction with the inclusion of aqueous extracts containing polyphenols from DDSP.

## 2. Materials and methods

### 2.1. Materials

The Al Foah date company, United Arab Emirates, supplied date seeds of Khalas variety. Ingredients for biscuit formulation were purchased from local markets in Al Ain. Chemicals and solvents were sourced from Fisher Scientific (Nepean, ON) and from Sigma–Aldrich Chemical Co. (St. Louis, MO).

### 2.2. Ultrasound-assisted extraction (UAE) and residue preparation

Finely ground DSP was sieved to obtain: (1) < 125 μm (small size) (2) 125 – 300 μm (medium size), and (3) 300–500 μm (large size) particles. Sieved powder was defatted with n-hexane for 2 h at 25 °C. UAE of the obtained powders was conducted according to [17] using an ultrasonic probe system (model VCX750, Sonics & Materials, INC., USA). Briefly, UAE was conducted with a solid to solvent ratio of 1:25 (w/v) for 8 min at 90 % amplitude. The resultant mixture was centrifuged and filtered to obtain the extract. Remaining residue was dried at 70 °C. Extract and residue were stored at −20 °C until further use.

### 2.3. Dough and biscuit preparation

For dough preparation, wheat flour (80 g), icing sugar (38 g), butter (13 g), vegetable oil (13 g), NaHCO3 (1.2 g), NaCl (1 g), milk powder (4 g), and water (19 mL) were used. First, oil and butter were mixed followed by icing sugar, milk powder and salt. After proper mixing of the above ingredients, wheat flour was added. Finally, the remaining ingredients were added, mixed well and kneaded. For the composite samples, water was replaced by the relevant extract [obtained from small (0.70 mg GAE/ ml water), medium (0.60 mg GAE/ ml water) and large (0.31 mg GAE/ ml water) particles] and wheat flour was substituted by the fiber-rich residue in three substitution levels; 7.5 %, 5 % and 2.5 % (w/w). Dough incorporated with fiber-rich residue obtained from small, medium and large sized particles of DDSP are denoted by S, M and L, respectively. Three more treatments were formulated which consisted of DDSP extracts obtained from S, M and L particles, instead of water with no substitution of fiber-rich residue.

Control sample contained neither the extract nor the residue. Some portions of all the dough samples were kept for analysis and the other parts were sheeted into uniform thickness of 3 mm for biscuit formulation. Using a stainless-steel circular mould, dough was cut into circles with 4.5 cm diameter. Biscuits were baked in an oven (Miele & Cie. KG, Bielefeld, Germany) at 180 ∘C for 12–14 min and then cooled at room temperature. Biscuits were packed in airtight packaging bags until further use. Dough and biscuit are denoted by the symbols D and B respectively. Table 1 presents different formulation of dough and biscuits and the acronyms used.

**Table 1**
Acronyms used for different samples and their explanation.

| Dough Sample | Acronym | Biscuit Sample | Acronym |
|---|---|---|---|
| 2.5 % substitution of residue + extract of small particles | 2.5SD | 2.5 % substitution of residue + extract of small particles | 2.5SB |
| 2.5 % substitution of residue + extract of medium particles | 2.5MD | 2.5 % substitution of residue + extract of medium particles | 2.5 MB |
| 2.5 % substitution of residue + extract of large particles | 2.5LD | 2.5 % substitution of residue + extract of large particles | 2.5LB |
| 5 % substitution of residue + extract of small particles | 5SD | 5 % substitution of residue + extract of small particles | 5SB |
| 5 % substitution of residue + extract of medium particles | 5MD | 5 % substitution of residue + extract of medium particles | 5 MB |
| 5 % substitution of residue + extract of large particles | 5LD | 5 % substitution of residue + extract of large particles | 5LB |
| 7.5 % substitution of residue + extract of small particles | 7.5SD | 7.5 % substitution of residue + extract of small particles | 7.5SB |
| 7.5 % substitution + extract of medium particles | 7.5MD | 7.5 % substitution + extract of medium particles | 7.5 MB |
| 7.5 % substitution of residue + extract of large particles | 7.5LD | 7.5 % substitution of residue + extract of large particles | 7.5LB |
| 0 % substitution of residue + extract of small particles | ESD | 0 % substitution of residue + extract of small particles | ESB |
| 0 % substitution of residue + extract of medium particles | EMD | 0 % substitution of residue + extract of medium particles | EMB |
| 0 % substitution of residue + extract of large particles | ELD | 0 % substitution of residue + extract of large particles | ELB |
| 0 % substitution of residue + water (control) | CD | 0 % substitution of residue + water (control) | CB |

### 2.4. Rheological behaviour

The rheological characteristics of the dough were analyzed utilizing a rheometer (Discovery Hybrid Rheometer, TA Instruments, Delaware, USA) equipped with parallel plate geometry (40 mm diameter) and an adjusted gap of 2 mm. Before testing the dough, sample was laid for 10 min to reach the thermal equilibrium and relax the stresses.

#### 2.4.1. Frequency sweep test

Prior to frequency sweep test, the linear viscoelastic range (LVR) was determined using oscillatory amplitude tests conducted at a temperature of 25 °C and a frequency of 1 Hz varying the strain value from 0.01 to 10 %. Frequency sweep tests were conducted at 25 °C within the frequency range from 1 to 20 Hz. Ratio of the measured G' and G″ was used to obtain the phase angle (tan δ) value.

#### 2.4.2. Creep and recovery test

At a temperature of 25 °C, creep recovery tests were performed ensuring the samples were in LVR. This involved applying a constant shear stress of 40 Pa for 100 s during the creep phase. Subsequently, the stress was released, and the strain was monitored during the 200 s recovery phase.

Creep-recovery data obtained can be presented as:

$$J(t) = \gamma(t) / \sigma$$

where J=compliance, γ = strain, and σ = constant stress.

The data obtained for creep and recovery phases were analysed using Burger's model.

$$\textit{Creep phase}: J(t) = J_i + J_r(1 - \exp(-t/\lambda)) + t/\eta_0.$$

$$\textit{Recovery phase}: J(t) = J_{max} - J_i - J_r(1 - \exp(-t/\lambda))$$

where $J_i$ = instantaneous compliance, $J_r$ = retarded compliance, $J_{max}$ = maximum compliance, λ = average retardation time and $\eta_0$ = Newtonian viscosity [29].

### 2.5. Textural properties

TPA of dough samples and the hardness of biscuit samples were performed using a texture analyzer (BROOKFIELD CT3, Brookfield Engineering Labs, Inc., Middleborough, MA, USA). The dough samples were compressed using a cylinder set probe (TA 11/1000). Test speed was 2 mm/s while the distance and the trigger force were 2.4 mm and 5 g, respectively. Evaluated texture parameters of dough were hardness, cohesiveness, springiness and adhesiveness. For biscuits, a sharp blade-cutting probe (TA7) was used with a test speed of 1 mm/s, a trigger load of 3.0 g and a distance of 10.0 mm.

### 2.6. Determination of colour attributes

The colour of the dough and biscuit samples were measured depending on L* (lightness), a* (red/green coordination) and b* (yellow/blue coordination), using a colourimeter (HunterLab ColourFlex EZ, Hunter Associates Laboratory, Inc., VA, USA).

### 2.7. Scanning electron microscopic (SEM) analysis

Microstructure of the dough and biscuit samples were investigated using a scanning electron microscope (SEM; JSM-6010PLUS/LA LA JEOL, Japan) with a voltage of 10 kV and x500/x600 magnifications.

### 2.8. Physical attributes of biscuit

Method followed by Mahloko et al. [30] was used to determine the thickness, spread ratio and diameter of biscuits, with slight modifications. To measure the diameter, three biscuit samples were placed edge to edge and the total diameter was measured. For thickness, six biscuit samples were placed on one another, and the total height was measured. The average diameter and thickness of a biscuits was recorded in millimetres. All the measurements were taken using a digital vernier caliper. Diameter and thickness values were used to calculate the spread ratio.

### 2.9. Statistical analysis

All the formulations of the dough and the biscuits were carried out in 3 batches and the analysis were performed in triplicates (except diameter, thickness, and weight of biscuit which were carried out in six analytical replicates). Analysis of variances (ANOVA) was used to analyse the data obtained, followed by Tukey's multiple comparison test to determine the significance ($P<0.05$) using Minitab 21 software.

## 3. Results and discussion

### 3.1. Rheological properties of dough

#### 3.1.1. Frequency sweep test

The rheological properties of dough can be used to forecast the behaviour of dough during processing and the quality of the resulting bakery product. Effect of incorporation of the extract and the fiber-rich extraction residue of DDSP on viscoelastic behaviour of dough in terms of small strain amplitude oscillatory analysis is presented in Fig. 1.

All dough samples displayed tan δ values less than 1 (tan δ < 1) within the frequency range tested, indicating the typical weak gel characteristics of wheat dough. Additionally, G' indicates elastic properties while G'' indicates viscous properties. As dough is viscoelastic in nature and tan δ = G''/ G' (which is < 1), higher G' values compared to G'' showed that elastic properties are predominant over viscous characteristics with solid-like behaviour. Changes in G' and G'' values of dough samples with frequency, are presented in supplementary material (**Figure S1**). Incorporation of the aqueous extract and the fiber-rich residue has resulted in a non-significant increase in the tan δ of the dough, compared to the control sample. Tan δ decreased with increasing the substitution level of fiber-rich residue with no clear difference between particle sizes (Fig. 1).

The graphs obtained for tan δ values of the dough samples showed almost a plateau above the frequency of 5 Hz (Fig. 1). The constancy of tan δ of dough was disturbed slightly with the incorporation of the extract and the fiber-rich residue of DDSP. Lower frequency range (up to 5 Hz) showed a decrease in tan δ indicating strong solid-like behaviour followed by an increase of tan δ within higher frequency range. Thus, dough samples showed a liquid like behaviour during rapid deformation [31] towards high frequency range. Overall, behaviour of the loss tangent of dough samples was the same within the given frequency range. Increase in tan δ values is directly linked to a decrease in the dominance of elasticity and an increase in the contribution of viscous components [32]. Increased tan δ values are also an indication of weakening of dough structure. However, the difference between the tan δ values of composite dough samples and the control sample was ~ 0.02 indicating that tan δ was not affected significantly ($P>0.05$) with the incorporation of the extract and the residue into dough. Both G' and G'' moduli of dough increased with the incorporation of the extract and the fiber-rich residue. The inclusion of phenolic extract and fiber impacts the viscoelastic properties of dough leading to increased moduli values and either decreased or unchanged tan δ values [31,33,34].

Increase in the mechanical properties of dough samples incorporated only with the extract (ESD, EMD and ELD), might be attributed to the prevailing bioactive compounds in the extract. Phenolic compounds usually show dual action in interrupting the gluten network, through dissociating sulphide bonds due to the antioxidant activity and then cross-linking with gluten network [35,36]. Date seeds contain a vast

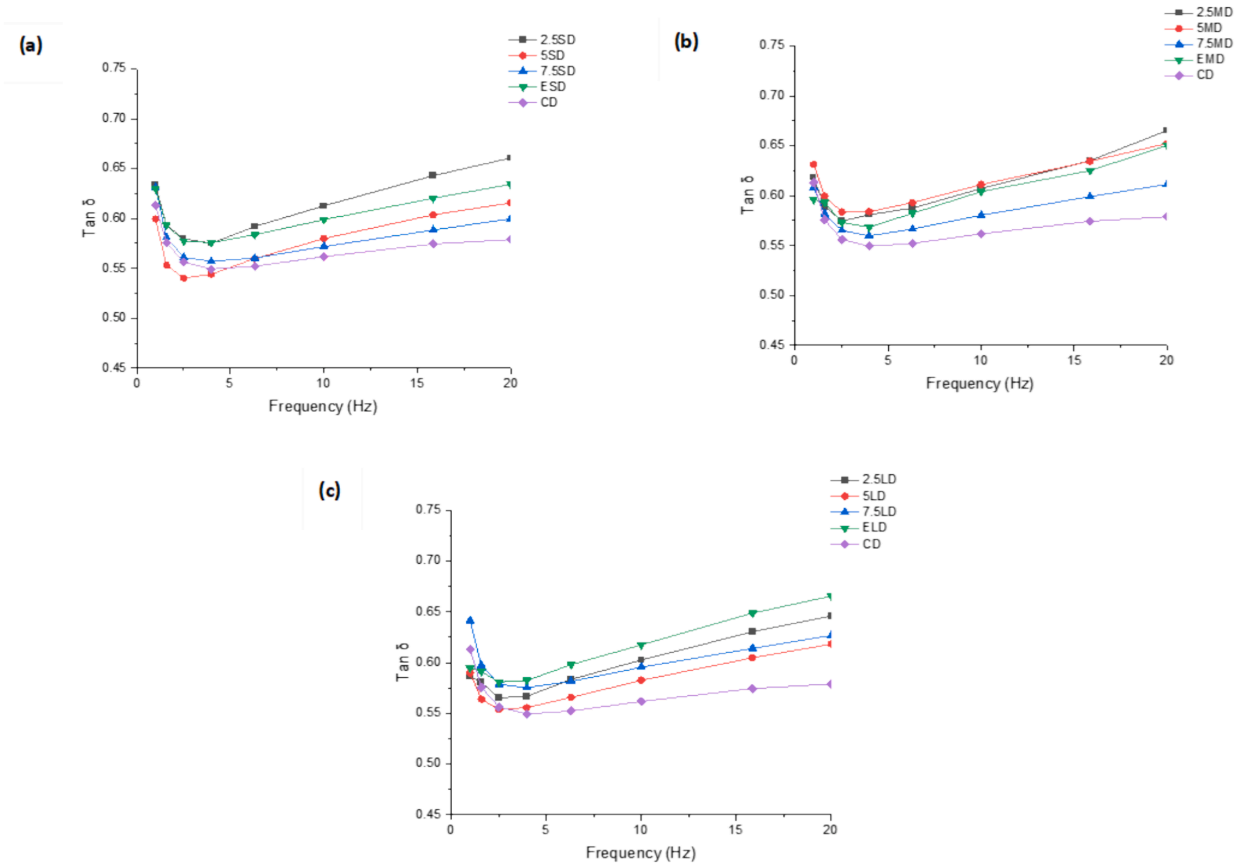


**Fig. 1.** Tan δ values of the control dough and the dough samples with different substitution levels of extraction residue obtained from UAE (2.5 %, 5 % and 7.5 %) (a) dough samples with small sized particles of the residue, (b) dough samples with medium sized particles of the residue and (c) dough samples with large sized particles of the residue. (T=25 °C).

amount of phytochemicals like phenolic acids, carotenoids, tocopherols and phytosterols [10]. The hydroxy phenyl residues and double bonds found in bioactive components of date seeds could potentially result in the formation of cross links with gluten. Besides, gluten proteins have a tendency to create intermolecular interactions with neighbouring proteins and starch molecules, further altering the rheological properties. Polyphenols have the potential for interference with S-H and S-S bonds and initiate depolymerization. Subsequently, repolymerize through hydrogen and hydrophobic bonds, instead of promoting disulphide bond formation [36,37]. Increased degree of intermolecular interactions results in more ordered and compact structure in gluten network having increased viscoelasticity with higher G' and G'' moduli values. Comparable findings were noted when the wheat dough was incorporated with green tea polyphenols, leading to an improvement in the dough's viscoelastic properties in terms of elasticity [37].

Within S particles, 5SD showed lower tan δ till 5 Hz and then it increased compared to CD (Fig. 1). Substitution of fiber-rich residue increased tan δ compared to the control, but with increasing substitution level a decrease in tan δ was observed. Within M particles, 2.5MD and 5MD concentration showed almost the same tan δ values. While for L particles, tan δ of 5LD was lower than 7.5LD. In summary, tan δ values decreased with increasing the substitution level of DDSP fiber-rich extraction residue, but with no significant difference ($P>0.05$) between particle sizes. G' and G'' moduli values were higher than the control sample in 5 % and 7.5 % substitution level of fiber-rich residue while at 2.5 % substitution level moduli were lower compared to control. Both moduli values decreased with increasing particle size. Increase in the complex modulus (G*) indicates that a material is showing more resistance to deformation. This was evident by the dynamic oscillation measurement results of the current study, with elevated G' and G'' values with the incorporation of fiber-rich extraction residue. Thus, it is evident that the extensibility of the dough became poor with the incorporation of the residue into dough. As evident by some previous studies, the incorporation of fiber significantly impacted the rheological aspects of dough [31,33,34,38]. Fiber can affect the viscoelastic properties of the dough in two ways. Most pronounced effect is the reduction of available water for gluten, affecting dough development. Dynamic moduli are greatly dependent on the water content of the gluten matrix [22]. The surface of fiber contains hydrophilic groups and a porous structure, which allows for the accommodation of more water molecules [39]. High water absorption capacity of fiber makes the gluten matrix dehydrated and restrict the mobility of particles within the matrix resulting a firm and rigid dough. Water acts as a plasticizer within the gluten matrix [31]. Due to the tight interaction of fiber with water, moisture distribution within the gluten matrix will be disturbed. Thus, the plasticizing effect will be weakened leading to an increase in G' and G'' moduli values. Additionally, fiber compounds have the potential to serve as fillers within the protein matrix. Decrease in water content occurs as a result of insoluble fiber, whereas soluble fiber can lead to a slight increase in viscosity [33]. This could potentially explain the slight rise in the tan δ value. Proportion of insoluble and soluble fiber plays a crucial role in deciding the effect on viscoelastic features of dough [40]. Fiber can strengthen the dough by improving the lubrication in gluten matrix due to its water absorption ability, promoting swelling of proteins and starch in dough system thereby increasing the tan δ [41]. Apart from the indirect influence through water absorption, fiber has the ability to directly interact with the side chain amino acids of proteins through hydrogen bonding [42]. Due to the composition of date seeds, there is a potential for interference with S-H bonds compared to S-S bonds, given the robust stability of disulphide bonds in gluten.

Moreover, insoluble fiber can be surrounded by starch granules making the dough structure inconsistent. The aforementioned phenomenon result in more rigid dough compared to the control dough [43] which might be a reason for the increased hardness in the dough observed in the current study. Studies have shown that the incorporation of fiber-rich powder obtained through extraction techniques such as ultrasound and microwave weakens dough, resulting in decreased resistance to deformation in cookie and bread dough, due to the increased water absorption capacity [44–47]. Ahmed et al. [44] observed the effects of incorporating date fiber, obtained through aqueous microwave extraction, into dough at levels of 2.5 %, 5 %, 7.5 %, and 10 %. Consistent with the results of the current study, the stiffness of the dough increased with fiber incorporation. However, in the present study, the 2.5 % substitution level had the least impact on the G' and G" moduli values, showing no significant difference compared to the control. This could be attributed to the changes in physical properties of DSP including the reduction in particle size, during ultrasonication [17,46] unlike microwave [48], which may decrease disruption to the gluten network, allowing for better blending with minimal effects on rheological properties of dough. The aforementioned observation suggests that fortifying biscuits with the fiber-rich fraction obtained through ultrasonication may offer greater feasibility for incorporation into biscuits compared to other extractions such as microwave.

G’ and G’’ moduli values increased with decreasing particle size except in 2.5 % substitution level of the fiber-rich residue. Increased surface area with decreased particle size allows more water molecules to accommodate compared to large particle sizes. Additionally, this results in more polymerization reactions apart from increased possibility of fiber molecules to have interactions with proteins and starch. Hence, enhanced viscoelastic properties were observed in small particle size of fiber-rich residue compared to large particles sized residue. The observed deviation in the 2.5 % substitution level, may be attributed to the more prominent physical barrier imposed by the large particles when the contribution of the residue to the dough development is minimal at the lowest substitution. Overall, dynamic oscillatory properties increased with decreasing particle size and increasing substitution level of the DDSP fiber-rich residue. Wheat dough incorporated with ginger powder [49] and grape peel powder [50] exhibited a similar behaviour as the addition levels increased with a subsequent increase in fiber content and decreasing particle size.

### 3.1.2. Creep and recovery test

Fig. 2 presents the impact of extract and fiber-rich residue of DDSP in terms of particle size and substitution level, on the mechanical properties of biscuit dough in creep and recovery phases.

Creep phase of the curve in all the samples, demonstrated non-linear deformation within the range where the strain increased over time. During the recovery period, samples exhibited the typical viscoelastic behaviour of a dough, demonstrating partial recovery in returning to their original state at extended durations. Aqueous extract incorporated dough samples (ESD, EMD and ELD) exhibited lower % strain values compared to the control with the lowest values in ESD. As mentioned in the section 3.1.1., polyphenols present in DDSP extract promote the depolymerization and polymerization reactions resulting in more intermolecular interactions within the gluten network making the structure of gluten more compact and ordered compared to the control.

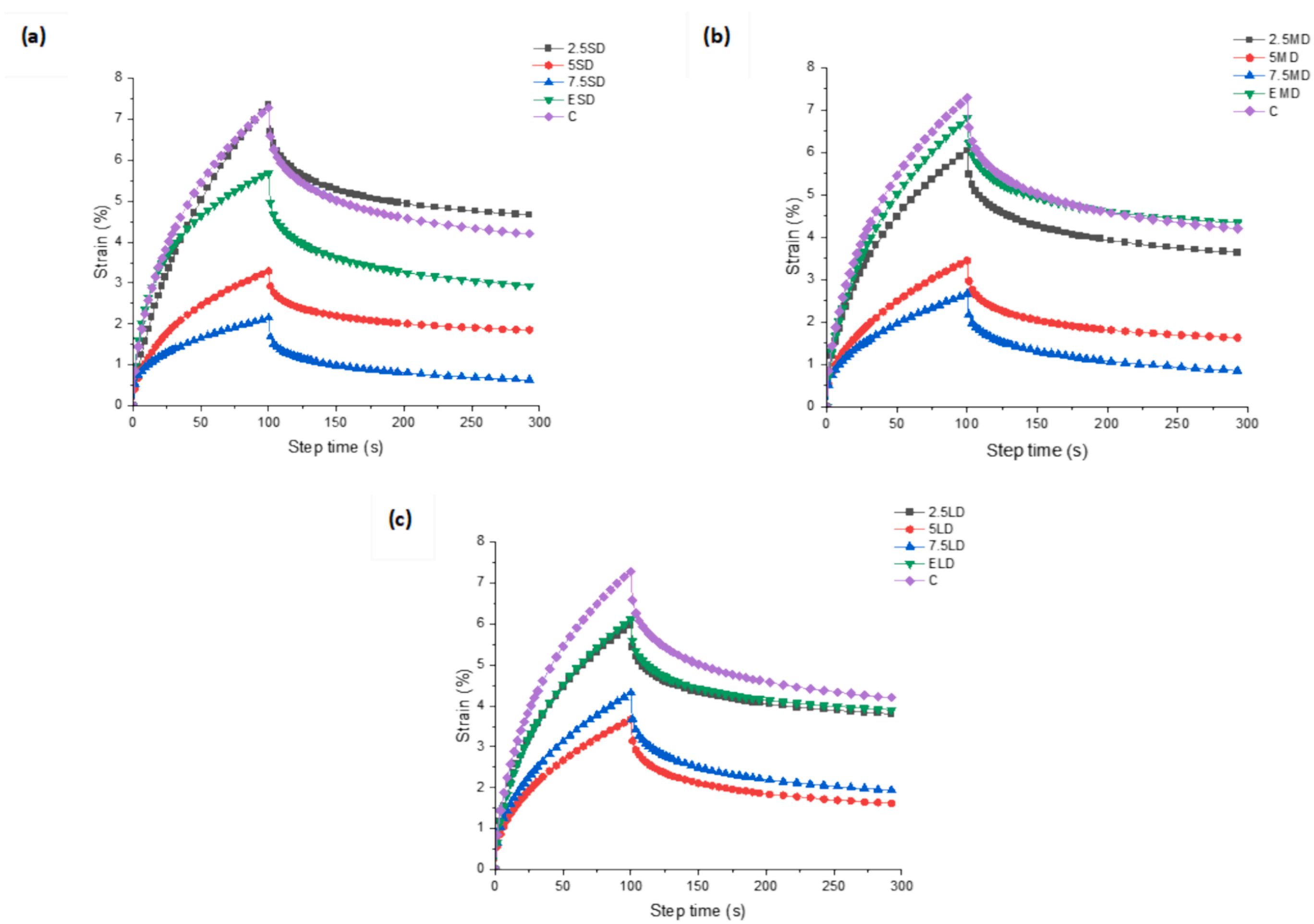


**Fig. 2.** Creep-recovery curves of the control dough and the dough incorporated with DDSP extraction residue obtained from UAE, (a) dough samples with small sized particles of DDSP, (b) dough samples with medium sized particles of DDSP and (c) dough samples with large sized particles of DDSP (T=25 °C).

This will result in increased strain values indicating increased resistance to deformation values with the added polyphenols [31]. Besides, with a low creep strain, the dough quickly returned back to the stable state. Due to high extraction efficiency in small particles with larger surface area, ESD contains a higher amount of polyphenols compared to EMD and ELD. Consequently, ESD incorporated sample exhibited lower strain values compared to EMD and ELD.

The maximum strain value obtained by the dough samples serves as an indication of the dough's rigidity [50]. In the creep phase, DDSP extraction residue incorporated dough exhibited minimal values for maximum creep achieved, indicating greater resistance to deformation, except in 2.5D which was similar to the control. When the substitution level of fiber-rich residue increased from 2.5 % to 7.5 % the capacity for deformation under applied stress became lower indicating lower creep compliances [51]. Large maximum creep strain values are an indication of high moisture content in gluten matrix. The high ability of fiber to accommodate more water molecules through hydrogen bonding enables increased competition between protein, starch and fiber for water interaction. The reduced availability of water for protein and starch results in decreased water distribution, and thereby reducing the lubrication of the dough matrix.

In L particle size, maximum creep compliance of 5 % substitution level was lower than the 7.5 % substitution level of fiber-rich residue with not much difference between the two samples. Resistance for deformation was increased with increasing particle size, which was evident by the increased maximum creep strain when the particle size increased. High strain values indicate better flow characteristics with increased softness whereas high recovery values suggest better elasticity [52]. Particle size significantly influences the elastic properties of dough, leading to a strong correlation between the particle size and the outcomes of the creep and recovery tests [50,53]. At the 7.5 % substitution level of fiber-rich residue, small particles showed a 70.51 % decrease in creep strain whereas in medium and large particle sized residue decreased the strain value by 63.37 % and 40.74 %, respectively. Increased surface area in small particles leads to more water absorption making the dough matrix more dehydrated compared to large particles. Effect demonstrated by large particles fiber-rich residue is mainly due to the physical disturbance to the structure, thus, affecting the consistency of the gluten matrix rather than low water absorption due to low surface area. This observation suggests that the effect on the water availability for dough development is predominant over the physical barrier imposed by the size of the particles of the residue. Cookie dough incorporated with grape peel powder exhibited a similar behaviour with decreasing particle size [50].

When the stress was removed (beginning of the recovery phase) all the samples showed an instantaneous decrease in strain response which is related to instantaneous compliance. The instantaneous compliance of a material is related to the elastic characteristics while the Newtonian compliance is related with viscous material which indicates the permanent breakage of the bonds [54]. The recovery strain decreased with relaxation, as the substitution level of the fiber-rich residue increased and particle size decreased. However, the rate of the decrease in strain values was found to less when the particle size decreased and and substitution level increased, indicating that fiber incorporation enhanced the solid elastic properties of the dough. This observation could be attributed to the enhancement of the dough's strain hardening capability by the incorporation of the fiber in the DDSP residue, which boosts the tensile strength of the gluten and consequently elevates the creep recovery rate of the dough [55]. This effect is further enhanced by the increased surface area in small particles, enabling a greater number of interactions between fiber and other components including water and protein. Overall, the viscoelastic properties of the dough increased with increasing levels of fiber-rich residue and decreasing particle size.

### 3.2. Textural properties

Textural attributes of the dough and biscuit samples are presented in Table 2 and Table 3, respectively. Hardness, springiness, cohesiveness and adhesiveness of ESD, EMD and ELD were not significantly different ($P>0.05$) from CD suggesting that addition of the polyphenolic extract did not affect on the textural properties of the dough. However, incorporation of the fiber-rich residue increased the hardness of the dough non-significantly ($P>0.05$), except 7.5MD and 7.5SD produced a significant increase ($P<0.05$) in hardness. Similarly, the springiness in the 7.5MD and 7.5SD samples were significantly lower ($P<0.05$) compared to CD whereas cohesiveness was significantly lower ($P<0.05$) in dough substituted with small particles at all three substitution levels. Furthermore, the adhesiveness was found to be significantly lower ($P<0.05$) in all the DDSP fiber-rich residue incorporated samples compared to CD. The increase in dough hardness and decrease in springiness, cohesiveness and adhesiveness at higher levels of residue substitution suggests that the addition of fiber is linked to the observed effects on texture profile of dough. The effects were prominent when the particle size of the residue decreased.. A prior study revealed that incorporating apple peel fiber led to an elevation in dough hardness and a reduction in adhesiveness, correlating with higher levels of inclusion and larger particle size [56]. Mironeasa et al. [50] found that the hardness of the dough increased, and the adhesiveness decreased with increase in the addition level and decrease in the particle size of grape peel powder, which is in agreement with our findings.

Similar trend was observed in biscuits where the hardness increased with elevated substitution levels and decreased particle size of fiber-rich residue. At the highest substitution level, samples added with all three particle sizes and 5SB exhibited a significant increase in biscuit hardness compared to CB. Within the same substitution level, small particles incorporated biscuits showed higher hardness compared to those with large particles ($P>0.05$). Comparable findings were observed with the cookies incorporated with blueberry and raspberry pomace fiber concentrates [57]. According to Yassin et al. [46], bread made with ultrasound-extracted sugarcane fiber showed a moderate firmness, which was lower than that of high-pressure homogenization but higher than that of microwave irradiation. Additionally, Najjar et al. [28] observed a significant impact on the texture with increasing substitution level of DSP in cookies. Similarly, biscuits incorporated with fiber-rich powder acquired after the aqueous extraction of the by-products of maize, showed enhanced hardness at the highest tested substitution level of 20 % with no significant difference between fine and coarse particles [58]. The fiber extracted through ultrasonication has demonstrated improved water holding and oil holding capacities by disrupting the crystal structure of fiber components [46,59,60]. These effects have clearly had a positive impact on the quality characteristics of cookies, as well as consumer acceptance when okara fiber was incorporated [45]. Ultrasonication generates pores on the surface of fiber molecules, while microwave extraction results in surface cracks [46]. As a result, the entrapment of molecules is higher in ultrasonicated fiber-rich powder compared to microwave-extracted powders [46]. In contrast to the findings of the present research, bread substituted with grape pomace fiber extracted using microwave exhibited reduced hardness, possibly due to the disruption of fiber component compactness [47]. The enhanced compactness of fiber resulting from ultrasonication might be attributed to the formation of uniform holes on fiber surface, resulting high water absorption and increasing the hardness.

High water absorption capacity in fiber makes the dough system dehydrated resulting to a firmer and drier dough compared to the control. Fiber interacts with protein disrupting the gluten-starch interactions and consequently impeding dough development. Impairment of the prevailing interactions within the dough matrix due to fiber-protein interactions [61] leads to a reduction in softness. This impact will be enhanced by the increased degree of cross-linking caused by fiber components, reducing the long polymer chains within gluten network

**Table 2**
Effect of incorporation of polyphenolic extract and fiber-rich extraction residue after UAE, on the textural properties and color attributes of biscuit dough.

| Sample | Hardness (N) | Springiness | Cohesiveness | Adhesiveness (mJ) | Colour | | |
|---|---|---|---|---|---|---|---|
| | | | | | L* | a* | b* |
| CD | 8.483 ± 0.42$^{c}$ | 1.54 ± 0.03$^{a}$ | 0.51 ± 0.00$^{a}$ | 0.91 ± 0.06$^{a}$ | 70.68 ± 0.52$^{a}$ | 2.89 ± 0.30$^{h}$ | 31.20 ± 1.38$^{a}$ |
| ESD | 8.971 ± 0.68$^{bc}$ | 1.31 ± 0.11$^{abc}$ | 0.45 ± 0.06$^{ab}$ | 0.450.07$^{bc}$ | 67.20 ± 1.40$^{a}$ | 4.06 ± 0.42$^{g}$ | 30.70 ± 0.47$^{a}$ |
| EMD | 8.31 ± 0.96$^{c}$ | 1.47 ± 0.07$^{ab}$ | 0.46 ± 0.01$^{ab}$ | 0.55 ± 0.04$^{b}$ | 68.12 ± 0.90$^{a}$ | 3.64 ± 0.35$^{gh}$ | 30.84 ± 0.29$^{a}$ |
| ELD | 8.75 ± 0.16$^{bc}$ | 1.300.04$^{abc}$ | 0.45 ± 0.03$^{ab}$ | 0.46 ± 0.09$^{bc}$ | 69.43 ± 1.88$^{a}$ | 2.84 ± 0.19$^{h}$ | 30.67 ± 0.32$^{a}$ |
| 2.5SD | 11.04 ± 0.34$^{abc}$ | 1.17 ± 0.01$^{abc}$ | 0.33 ± 0.02$^{bc}$ | 0.35 ± 0.06$^{bc}$ | 49.31 ± 1.25$^{ef}$ | 7.94 ± 0.53$^{cd}$ | 18.08 ± 0.16$^{e}$ |
| 2.5MD | 10.85 ± 1.41$^{abc}$ | 1.18 ± 0.04$^{abc}$ | 0.39 ± 0.01$^{ab}$ | 0.42 ± 0.04$^{bc}$ | 55.75 ± 1.34$^{cd}$ | 6.60 ± 0.10$^{e}$ | 21.54 ± 0.12$^{c}$ |
| 2.5LD | 9.09 ± 0.23$^{bc}$ | 1.16 ± 0.06$^{abc}$ | 0.42 ± 0.1$^{ab}$ | 0.47 ± 0.06$^{bc}$ | 60.15 ± 0.99$^{b}$ | 5.63 ± 0.04$^{f}$ | 24.68 ± 0.67$^{b}$ |
| 5SD | 13.54 ± 3.74$^{abc}$ | 1.14 ± 0.09$^{bc}$ | 0.28 ± 0.06$^{bc}$ | 0.36 ± 0.04$^{bc}$ | 42.88 ± 1.07$^{g}$ | 9.03 ± 0.14$^{ab}$ | 13.81 ± 0.43$^{f}$ |
| 5MD | 9.87 ± 0.58$^{abc}$ | 1.17 ± 0.04$^{abc}$ | 0.37 ± 0.01$^{abc}$ | 0.35 ± 0.01$^{bc}$ | 50.59 ± 1.52$^{e}$ | 7.39 ± 0.20$^{de}$ | 17.59 ± 0.84$^{e}$ |
| 5LD | 9.24 ± 0.53$^{bc}$ | 1.17 ± 0.27$^{abc}$ | 0.41 ± 0.1$^{ab}$ | 0.40 ± 0.01$^{bc}$ | 57.07 ± 1.01$^{bc}$ | 6.80 ± 0.43$^{e}$ | 20.59 ± 0.72$^{cd}$ |
| 7.5SD | 17.78 ± 3.09$^{a}$ | 1.01 ± 0.00$^{c}$ | 0.21 ± 0.01$^{c}$ | 0.31 ± 0.11$^{c}$ | 37.51 ± 0.60$^{h}$ | 9.34 ± 0.26$^{a}$ | 11.26 ± 0.66$^{g}$ |
| 7.5MD | 16.87 ± 4.95$^{ab}$ | 1.11 ± 0.13$^{bc}$ | 0.35 ± 0.01$^{abc}$ | 0.34 ± 0.04$^{bc}$ | 45.73 ± 3.27$^{fg}$ | 8.41 ± 0.34$^{bc}$ | 14.89 ± 1.03$^{f}$ |
| 7.5LD | 11.15 ± 1.43$^{abc}$ | 1.22 ± 0.08$^{abc}$ | 0.42 ± 0.01$^{ab}$ | 0.39 ± 0.05$^{bc}$ | 52.36 ± 0.62$^{de}$ | 7.34 ± 0.10$^{de}$ | 18.73 ± 0.78$^{de}$ |

Means ± SD are presented. Different lowercase superscript letters in a column denote significant differences, $P < 0.05$.

**Table 3**
Effect of incorporation of polyphenolic extract and fiber-rich extraction residue after UAE on the textural properties and color attributes of biscuits.

| Sample | Hardness (N) | Diameter (mm) | Thickness (mm) | Spread ratio | Colour | | |
|---|---|---|---|---|---|---|---|
| | | | | | L* | a* | b* |
| CB | 15.16 ± 0.77$^{d}$ | 54.39 ± 0.15$^{ab}$ | 5.34 ± 0.21$^{abc}$ | 10.19 ± 0.40$^{ab}$ | 69.91 ± 1.26$^{a}$ | 8.99 ± 0.86$^{ab}$ | 37.40 ± 1.17$^{a}$ |
| ESB | 16.02 ± 2.36$^{cd}$ | 53.50 ± 0.80$^{abc}$ | 5.29 ± 0.03$^{bc}$ | 10.105 ± 0.17$^{abc}$ | 68.56 ± 2.06$^{a}$ | 8.94 ± 0.88$^{ab}$ | 35.46 ± 0.20$^{ab}$ |
| EMB | 15.71 ± 1.67$^{d}$ | 53.34 ± 0.81$^{abc}$ | 5.45 ± 0.18$^{abc}$ | 9.79 ± 0.23$^{abcd}$ | 66.76 ± 0.99$^{ab}$ | 10.39 ± 0.57$^{ab}$ | 35.87 ± 0.02$^{ab}$ |
| ELB | 15.55 ± 1.59$^{d}$ | 54.90 ± 0.32$^{a}$ | 5.09 ± 0.03$^{c}$ | 10.79 ± 0.12$^{a}$ | 69.46 ± 0.62$^{a}$ | 8.99 ± 0.42$^{ab}$ | 36.27 ± 0.83$^{ab}$ |
| 2.5SB | 18.55 ± 1.19$^{abcd}$ | 54.81 ± 0.35$^{a}$ | 5.25 ± 0.11$^{bc}$ | 10.45 ± 0.25$^{a}$ | 59.56 ± 1.22$^{d}$ | 8.96 ± 0.03$^{ab}$ | 28.02 ± 1.03$^{ef}$ |
| 2.5 MB | 16.24 ± 2.84$^{bcd}$ | 54.71 ± 0.68$^{a}$ | 5.18 ± 0.67$^{bc}$ | 10.68 ± 01.33$^{a}$ | 61.08 ± 0.71$^{cd}$ | 10.34 ± 0.59$^{ab}$ | 31.29 ± 0.56$^{cd}$ |
| 2.5LB | 16.20 ± 1.45$^{bcd}$ | 54.92 ± 0.40$^{a}$ | 5.12 ± 0.09$^{c}$ | 10.73 ± 0.13$^{a}$ | 64.58 ± 1.17$^{bc}$ | 9.00 ± 0.72$^{ab}$ | 33.39 ± 0.86$^{bc}$ |
| 5SB | 22.65 ± 0.84$^{ab}$ | 52.06 ± 0.87$^{bcd}$ | 5.27 ± 0.12$^{bc}$ | 9.89 ± 0.33$^{abcd}$ | 54.33 ± 0.05$^{e}$ | 8.72 ± 0.86$^{b}$ | 22.82 ± 1.34$^{g}$ |
| 5 MB | 17.48 ± 1.50$^{abcd}$ | 52.53 ± 1.70$^{abcd}$ | 5.37 ± 0.37$^{abc}$ | 9.81 ± 0.61$^{abcd}$ | 59.93 ± 0.13$^{d}$ | 8.92 ± 0.02$^{ab}$ | 28.10 ± 0.15$^{ef}$ |
| 5LB | 16.62 ± 0.66$^{abcd}$ | 51.46 ± 0.90$^{cd}$ | 5.76 ± 0.20$^{abc}$ | 8.94 ± 0.16$^{bcde}$ | 60.66 ± 1.08$^{cd}$ | 9.73 ± 0.82$^{ab}$ | 31.35 ± 0.88$^{cd}$ |
| 7.5SB | 23.08 ± 2.11$^{a}$ | 51.69 ± 0.95$^{cd}$ | 5.90 ± 0.10$^{ab}$ | 8.76 ± 0.29$^{cde}$ | 51.08 ± 0.16$^{e}$ | 10.14 ± 0.13$^{ab}$ | 21.93 ± 0.32$^{g}$ |
| 7.5 MB | 22.29 ± 1.74$^{abc}$ | 51.21 ± 1.26$^{cd}$ | 5.93 ± 0.25$^{ab}$ | 8.65 ± 0.16$^{de}$ | 52.91 ± 0.96$^{e}$ | 11.20 ± 0.27$^{a}$ | 27.08 ± 0.54$^{f}$ |
| 7.5LB | 22.60 ± 1.17$^{ab}$ | 50.61 ± 0.88$^{d}$ | 6.05 ± 0.04$^{a}$ | 8.36 ± 0.11$^{e}$ | 61.39 ± 0.39$^{cd}$ | 9.00 ± 0.45$^{ab}$ | 30.25 ± 0.91$^{de}$ |

Means ± SD are presented. Different lowercase superscript letters in a column denote significant differences, $P < 0.05$.

[61]. Cross-linking can be further enhanced by the phenolic components present in the water extract of DDSP [31]. Increased abundance of fiber in the highest substitution level resulted in significant differences in textural properties of composite samples compared to control. Furthermore, increased surface area in particles with small size allows fiber to make more interactions heightening the effects. Thus, hardness of dough and biscuit samples increased while the springiness, cohesiveness and adhesiveness of the dough samples decreased with decreasing particle size and increasing substitution levels of DDSP fiber-rich residue. Hence, it is essential to consider the quantities of extracted materials and water content to be incorporated, for achieving the desired physical quality of baked goods, which may also vary based on the type of bakery item being prepared.

### 3.3. Colour attributes of dough and biscuits

Changes in color attributes of the dough and biscuit samples as affected by incorporation of extract and fiber-rich residue from DDSP is presented in Table 2 and Table 3. There was no significant difference (P>0.05) in colour attributes observed between the samples incorporated only with the polyphenolic extract and the control sample, both in the dough and the biscuits. However, there was a decrease in L* and b* of the dough and biscuits, with the incorporation of fiber-rich residue. As the substitution level of the residue increased and the particle size decreased, both the dough and biscuits became darker. a* of the dough increased with increasing substitution level and decreasing particle size of fiber-rich residue, whereas there was no significant difference (P>0.05) between the control and residue incorporated samples of biscuit.

The darker colour of the dough and biscuits with the incorporation of the fiber-rich residue could be attributed to the water-insoluble phenolic compounds and the fiber components present in the extraction residue after UAE. Phenolic compounds retained in the residue have the ability to enhance the enzymatic browning resulting in the decreased yellowness and lightness [62]. Because of the pigments like tannins present, the DDSP extraction residue itself has a dark brown colour which reduces the lightness in wheat flour. Prior research has indicated that the incorporation of DSP results in a significant darkening of bakery products such as cookies and bread [28,63] which are in line with the current study. Direct substitution of DSP in cookies resulted L* value of 40.55–48.32 [28] while in the current study L* varied between 51.08–64.58. Thus, compared to the direct addition of DSP, incorporation of DDSP fiber-rich residue obtained through UAE along with the extract, enhances the quality of biscuits in terms of colour.

The presence of sugars and proteins in dough leads to the Maillard and caramelization reactions while baking biscuits at high temperatures, hence the biscuits becomes darker compared to the dough. The occurrence of the Maillard reaction may account for the increase in redness (a*) of biscuits observed with the incorporation of fiber-rich residue with no significant difference (P>0.05) between the composite and control biscuits. The inclusion of fiber can facilitate the interaction between sugars and proteins in dough matrix by reducing the available water for their interaction. This process enhances the Maillard reaction, resulting in a darker colour change compared to the control sample [63]. Due to their larger surface area, small particles can interact and distribute more uniformly within the dough than large particles. Consequently, small particles have a greater influence on the dough's colour compared to large particles, as evident by the effect of particle size of the extraction residue on the colour parameter.

### 3.4. Dimensional characteristics of biscuits

Physical characteristics of biscuits incorporated with polyphenolic extract and fiber-rich residue are presented in Table 3. Diameter of biscuits with added polyphenolic extract (ESD, EMD and ELD) did not show any significant difference from the control biscuit (CB). When the biscuits were incorporated with fiber-rich residue, all three particle sizes within 7.5 % substitution level and 5LB samples exhibited significant decrease ($P<0.05$) in diameter compared to CB. Within one substitution level there was no significant difference ($P>0.05$) in diameter and spread ratio among particle sizes, but large particles showed lower diameter compared to small particles. Moreover, there was no significant difference ($P>0.05$) in thickness of the biscuit samples incorporated with extract and fiber-rich residue, while the biscuits with 7.5 % substitution of fiber-rich residue exhibited significantly higher spread ratio ($P<0.05$) compared to the CB.

Results of the current study suggests that diameter and the spread ratio of the biscuits decreases with increasing fiber content and particle size. Expansion of dough due to leavening process, flow of dough during baking as well as the set time could influence the spread ratio of biscuits [64]. The changes in spread ratio due to the incorporation of fiber-rich residue suggests that the components of the residue interact with dough components, influencing dough structure and development, as confirmed by the rheology results, thus causing reduction in the diameter and spread ratio of the biscuits. The reduction in these dimensional properties of biscuits could be attributed to the increased fiber content resulting from the substitution of fiber-rich extraction residue. The effect of incorporating fiber-rich components on the thickness, spread ratio and diameter of biscuits has been studied previously [26,62,65]. The presence of fiber leads to a decrease in the available water for proteins and starch in the dough impacting dough development. The presence of fiber will disrupt the development of a robust gluten network by inserting itself between the gluten molecules, thereby weakening the dough and hindering the capture of CO2. Thus, the leavening process in the dough is impeded, leading to a decrease in both the diameter and the spread ratio of biscuits [65]. Comparable outcomes were observed in bread that had 5 % microwave-extracted grape pomace fiber substituted, resulting in a decrease in specific volume [47]. In the present study, the inclusion of a 5 % fiber-rich fraction obtained after ultrasound extraction did not exhibit significant variances from the control, indicating that ultrasonicated powder has a lesser impact on the dough leavening process compared to microwave-extracted powder. However, additional research is warranted to compare the effects of extraction methods, as the results are greatly influenced by the type of baked goods.

The spread ratio is usually constrained by viscosity, which in turn is influenced by the fiber content [66]. Saeed et al. [26] has suggested that due to the increased hydrophilic sites within dough system, water mobility increases and hence viscosity increases, reducing the spread ratio. However, reduction of gluten content due to the substitution of DDSP extraction residue had shown a clear impact which was significant with the high substitution levels of the extraction residue. Furthermore, reduction of diameter and spread ratio in large particles was higher than in small particles. This suggests that physical barrier imposed by large particles on dough development is predominant over the chemical interference by fiber components. Substitution of peach and date powders in cookies [65] as well as DSP in biscuits [26] resulted in decrease of diameter and spread ratio. Aligning with the observations of the current study, biscuits incorporated with fiber and polyphenols rich mango peel powder showed decreased diameter and spread ratio [62]. Cookies and biscuits with high spread ratios are usually preferred as a desirable quality attribute [66,67]. On the other hand, decreased spread ratio improves the suitability for rotary mold preparation, where a reduced spread was preferred for a uniform embossing [26]. Therefore, it is crucial to regulate both the quantity and particle size of fiber-rich residue incorporated into biscuit, to achieve acceptable dimensional attributes while enhancing its functional properties.

### 3.5. Microstructural changes of the dough and biscuits with the incorporation of DDSP

Micrographs of wheat flour, ultrasonicated DDSP residue, control samples of dough and biscuit, and the dough and biscuit samples of 7.5 % substitution level with small particle size, are shown in Fig. 3. Microstructural images of wheat flour revealed the existence of irregular shaped fragments with different sizes which are likely to be the broken endosperm, appearing as clusters within gluten matrix. Additionally, several cellular components were observed, predominantly various sized starch granules attached to protein matrix (Fig. 3**a**). Raw DSP contains a regular, smooth and intact surface morphology [68,69] in which the cellulose fibers are covered by hemicellulose and lignin [70]. As a result of cavitation during ultrasonication, the surface of DDSP has undergone physical break down, resulting crevices and microfractures. The fibrous nature of DDSP has become prominently visible with hollow morphology, following the physical impact on the surface, imposed by ultrasonication (Fig. 3**b**). Jadhav et al. [68] observed comparable micrographs in their analysis of ultrasonicated DSP, aligning with the findings of the present study. In a prior study examining the impact of ultrasonication, microwave treatment, and high-pressure homogenization on sugarcane fiber incorporated into bread dough, it was noted that ultrasonication resulted in uniformly distributed holes on the fiber surface. In contrast, microwave treatment and high-pressure homogenization led to the observation of slight cracks and cracks with holes on the fiber surface, respectively [46].

Micrographs of CD exhibited a smooth, continuous and intact structure confirming the formation of a uniform, solid gluten network (Fig. 3**c**). Incorporation of fiber-rich residue resulted in a heterogeneous aggregation within the dough matrix (Fig. 3**d**). Large and small chunks, probably the starch granules were exposed outside the gluten network. This might be linked to a reduction in starch-protein interactions, resulting a poor and discontinues gluten network influenced by the presence of fiber. Otherwise, these aggregates could be linked with fiber components. Similar results were observed in dough samples incorporated with fiber rich components like wheat bran and mushroom fiber [41,71]. In line with the same pattern, smooth, continuous and well-developed protein network observed in the CB (Fig. 3**e**) was disrupted by the fiber-rich residue as indicated by Fig. 3**f**. Small aggregates were visible, embedded in the gluten matrix, resulting a heterogeneous surface in the composite biscuits. The SEM findings indicated that incorporation of DDSP fiber-rich residue disrupted the structure and the consistency of the gluten network, leading to a firm dough and biscuit, aligning with the rheological results.

## 4. Conclusion

The incorporation of the aqueous extract and fiber-rich extraction residue obtained through UAE had a significant impact on the rheological properties of the dough. G' and G″ increased with decreasing particle size and increasing substitution level while tan δ reduced with rising substitution level of fiber-rich extraction residue. The smallest particle size with the highest substitution level of the fiber-rich residue had dominant effect on the dough's viscoelastic attributes, while polyphenolic extract incorporation resulted to an increase in viscoelastic properties of the dough. Hardness of both the dough and the biscuits increased with decreasing particle size and increasing substitution level of the residue. Incorporation of the fiber-rich residue resulted darker dough and biscuits compared to the control samples. SEM images showed that the gluten network's continuity was disrupted due to the inclusion of the DDSP fiber-rich residue. Therefore, this study provides a valuable insight on utilizing date industry by-products, specifically

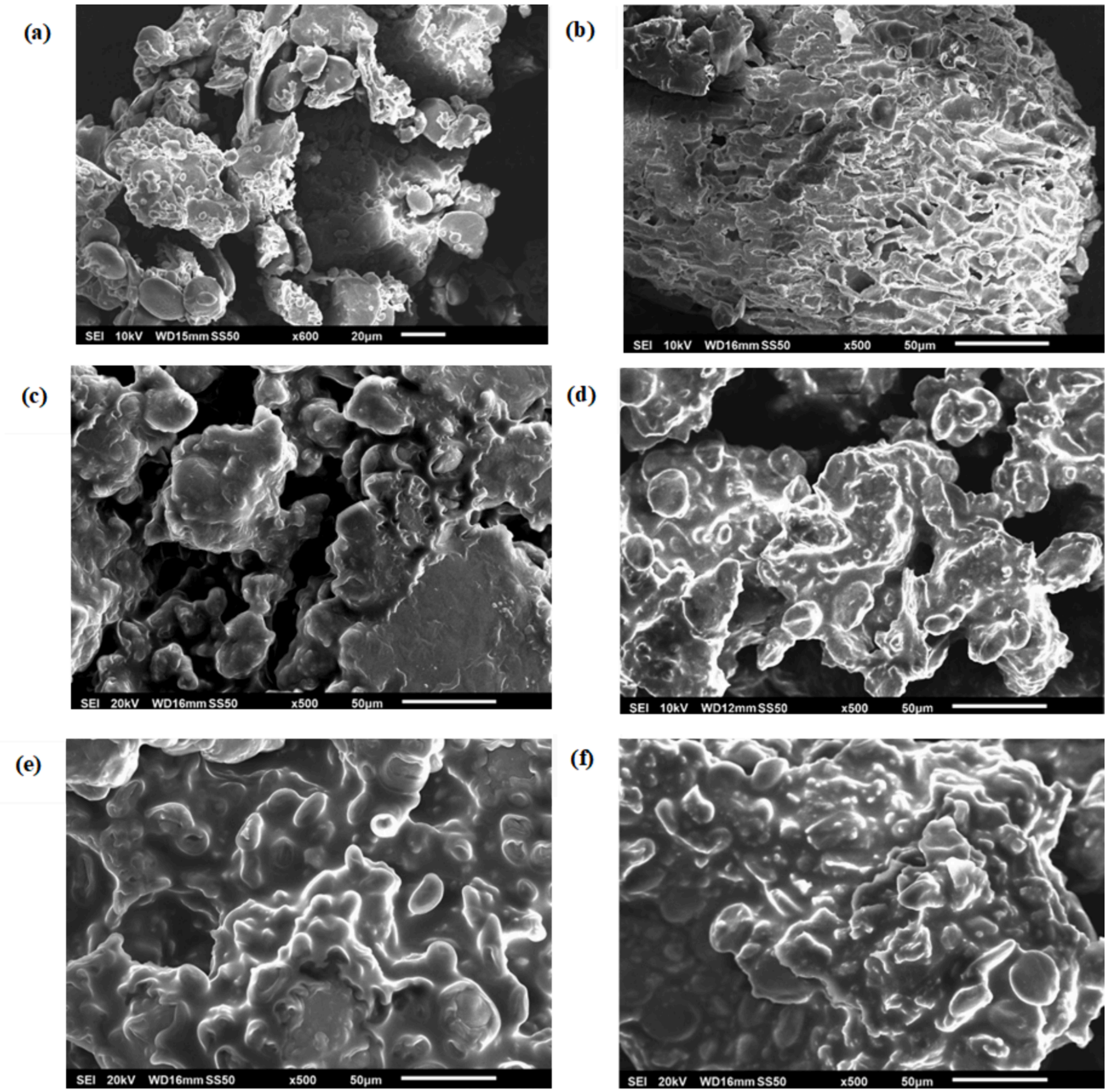


**Fig. 3.** Scanning electron microscopy (SEM) images of wheat flour (a), DDSP extraction residue obtained after UAE (b), control dough [CD] (c), control biscuit [CB] (d), dough [7.5SD] (e) and biscuit [7.5SB] (f) of small particles with 7.5% substitution level of fiber-rich residue.

seeds, to formulate functionally and physically improved bakery products through ultrasound technology towards a circular bioeconomy.

## CRediT authorship contribution statement

**Meththa Ranasinghe:** Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation. **Constantinos Stathopoulos:** Writing – review & editing, Supervision, Conceptualization. **Balan Sundarakani:** Funding acquisition, Project administration. **Sajid Maqsood:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

## Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Acknowledgement

Authors would like to acknowledge the funding received by the United Arab Emirates Ministry of Education; Project ‘READY’ [grant number G00003563]. Authors extend their thanks to Dr. Fathalla Hamed, Professor, Department of Physics, United Arab Emirates university for facilitating scanning electron microscopy analysis.

## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2024.107015.

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