



Article

Effects of Dry Heat Treatment and Milling on Sorghum Chemical Composition, Functional and Molecular Characteristics

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Abstract: This study aimed to highlight the effects of grains dry heat treatment, flour particle size and variety on sorghum flour nutritional, functional, and molecular characteristics. The results obtained showed that dry heat treatment led to fat, fiber and water absorption capacity increase, while the moisture, protein, ash, water retention capacity, solubility index, foaming capacity, and FT-IR absorption bands characteristic to phytic acids decreased with temperature applied raised. Particle size reduction determined lower protein, solubility index, and emulsifying activity and higher fat content, oil absorption capacity, swelling power, and foaming capacity. White sorghum flour fractions presented lower protein content, except when they were treated at 140 °C, lower carbohydrates and fibers and higher fat content compared to those made of red sorghum. Moderate significant correlations (p < 0.05) were observed between some of the functional properties and proximate composition of flours. Thus, both dry heat treatment conditions and particle size exerted significant influences of sorghum flour chemical and functional properties. These results showed the importance of particle size and dry heat treatment on sorghum flours functionality, being helpful for further optimizations and choices for bakery products use.

Keywords: sorghum flour; roasting; proximate composition; functional properties; particle size; FT-IR spectra



Citation: Batariuc, A.; Ungureanuluga, M.; Mironeasa, S. Effects of Dry Heat Treatment and Milling on Sorghum Chemical Composition, Functional and Molecular Characteristics. *Appl. Sci.* **2021**, *11*, 11881. https://doi.org/ 10.3390/app112411881

Academic Editor: Antonio Valero

Received: 5 November 2021 Accepted: 12 December 2021 Published: 14 December 2021

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1. Introduction

Sorghum (Sorghum bicolor (L.) Moench) is the fifth most cultivated cereal in the world, after corn, wheat, rice and barley [1], being a staple food crop in the semi-arid tropical regions of Africa, Asia, and Latin America [2]. Originally from East Africa (Sudan and Ethiopia), it has been cultivated all over the world due to its agronomic advantages, being resistant to drought, pests, and diseases, and flexible in planting time [3].

Sorghum is a small spheroid grain of approximately 3 mm in diameter that includes the germ and the endosperm inside the grain and the outer layers which give the red, brown, white, or black color [4]. Sorghum nutritional profile contains carbohydrates (54.6–77.2%), dietary fiber (4.5–26.3%), proteins (4.7–19.0%), and lipids (1.6–5.0%) [5–7]. Sorghum grains are also a good source of minerals (phosphorus, zinc, iron, calcium, magnesium, and potassium), B vitamins, vitamin E, β -carotene, and bioactive compounds (polyphenols and anthocyanins) [5,8]. Their high nutritional value varies depending on variety, cultivation region, and pericarp color [9]. Sorghum's outstanding nutritional properties make it a promising functional ingredient that offers the opportunity to produce foods with high levels of dietary fibers and antioxidants, and also with various natural colors [10,11] due to the specific pigment of each variety.

Appl. Sci. 2021, 11, 11881 2 of 17

Different food products such as breads [12,13], tortillas [14,15], snacks [16], pasta [17] and noodles [18] were produced using sorghum flour as functional ingredient. In addition, due to its content in resistant starch and glucans, sorghum can be used as a prebiotic food ingredient [19]. Being deficient in gluten protein, sorghum grain can supply the needs of people with gluten intolerance [1,20], while also being suitable for patients with diabetes and cardiovascular diseases [21].

During roasting, a variety of changes can inevitably occur and it is thus necessary to control the roasting time and temperature to obtain optimal characteristics. Roasted cereal grains may influence milling yield since they are generally characterized by decreased kernel hardness, due to the increased internal porosity of the endosperm [22]. Thermal changes induced by roasting impact kernel hardness by making it softer due to the loss of endosperm structure [23]. The roasting method and conditions under which processing is performed impact the product properties [24]. As Schoeman et al. [24] states, roasting could potentially serve as a pre-processing step to enable the use of less energy for milling, to produce value-added products, or to extend the shelf life of products. Sorghum flour sieving to different particle sizes might be a necessary processing step, taking into account to improve the quality of certain food. Dayakar Rao et al. [25] affirmed that biscuits produced from sorghum flours of 180 and 251 µm particle sizes had a better consumer acceptance than those produced from flours with smaller particle sizes (below 180 µm). In contrast, 60% sorghum extraction flour used to produce gluten-free bread showed a higher specific volume and softer texture than gluten-free bread from whole-grain sorghum flour [26]. On the other hand, transferring results research from lab-scale mills to an industrial scale milling system represents a challenge. Recently, Rumler et al. [27] investigated the effectiveness of sorghum milling when using two different milling systems and showed their impact on the chemical and physical properties of flour fractions and whole sorghum flours obtained.

Flour particle size and composition affect the functionality of the sorghum flour such as water absorption capacity, water solubility index, swelling power, pasting properties, and product quality [26,28,29]. The particle size is frequently associated with the surface area available for enzymatic action [29]. The sorghum cultivar and climatic conditions impact grain hardness [30]. Moreover, the sorghum cultivar determines the amylose/amylopectin ratio, which influences the extent of gelatinization and retrogradation, higher amylose content promoting retrogradation [18], A decrease in starch gelatinization properties was found due to the presence of kafirins, storage proteins with high hydrophobicity [31]. In addition, these proteins, stabilized by disulfide bonds, determined and a decrease in starch and protein digestibility due to the tight starch-protein matrix formed [32]. These undesirable aspects can be diminished by using thermal treatment such as roasting. Additionally, this processing method can decrease the antinutritional components found in raw sorghum grains (e.g., tannins, phytic acid, and protein cross-linker) [33,34] which reduces the feed efficiency [8], improving thus their nutritional value and products. In comparison with other grains, sorghum develops a pleasant taste and crispiness after roasting, a treatment that extends the shelf-life and safety of products by lowering the water activity [34]. Several studies have been undertaken to investigate the effects of roasting as a pre-milling treatment of sorghum grains. Ranganathan et al. [35] found that roasting of sorghum grains increased the water absorption capacity, offering beneficial effects for the preparation of instant mixes, porridge, and soup. Roasted sorghum flour exhibited better functional, pasting, and antioxidant properties when microwave processing was applied [36].

The aim of the present study was to investigate the effects of dry heat treatment of two sorghum varieties (red and white) on the functional, chemical composition, and molecular characteristics of flours with different particle sizes. For this purpose, the proximate composition of flours in terms of proteins, fat, carbohydrates, moisture, and ash were determined along with the molecular structures and functional properties in terms of hydration capacity, water absorption capacity, oil absorption capacity, solubility index, water retention capacity, swelling power, bulk density, emulsifying and foaming properties.

Appl. Sci. **2021**, 11, 11881 3 of 17

2. Materials and Methods

2.1. Sorghum Treatment

White sorghum grains (ES Albanus hybrid) were purchased from the Secuieni Agricultural Development Research Station (Neamṭ, România) and red sorghum grains (ES Alize hybrid) were purchased from a farmer (Suceava, România).

Dry heat treatment of sorghum grains at different temperatures (121 $^{\circ}$ C—T1 and 140 $^{\circ}$ C—T2) was done for 15 min in a Binder ED53L convection oven (Binder, Tuttlingen, Germany). Untreated sorghum grains were considered as control samples.

Integral sorghum flours (I) were obtained by grains milling with a laboratory grinder (Grain Mill, KitchenAid, Model 106 5KGM, Benton Harbor, MI, USA). In order to obtain sorghum flour at three different particle sizes, large (L > 300 μm), medium (200 $\mu m < M < 250~\mu m$), small fractions (S < 200 μm), the integral flour was sieved in a Retsch Vibratory Sieve Shaker AS 200 basic (Haan, Germany).

2.2. Proximate Composition

The nutritional composition (moisture, protein, fat, ash) of sorghum flours were analyzed using ICC methods: moisture (101/1), fat (104/1), protein (105/2), and ash (105/1). Total dietary fiber was determined by using Megazyme kit (K-TDFR-200a 04/17), according to the AACC 32-05.01 method. The carbohydrate content was calculated by difference, by applying the equation [37] (Equation (1)):

Carbohydrates (%) =
$$100 - (protein + fat + ash + fiber + moisture)$$
 (1)

The energetic value (kcal/100 g) of the samples was also calculated by multiplying nutrients values by their corresponding conversion coefficients [37] (Equation (2)):

Energy (kcal/100 g) =
$$(4 \times \text{protein}) + (9 \times \text{fat}) + (4 \times \text{carbohydrates}) + (2 \times \text{fiber})$$
 (2)

2.3. Functional Properties of Sorghum Flours

2.3.1. Hydration Capacity

The hydration capacity was determined in duplicate, according to the method described by Bordei [38]. For this purpose, 5 g of flour were weighed into a 50 mL tube and 30 mL of tap water was added. After mixing with a rod for 30 s at 10 min intervals for 1 h, the rod was washed over the tube with 10 mL of water and the suspension was centrifuged for 20 min at 2300 rpm. After removal of the supernatant, the sample was kept at 50 °C for 25 min and weighed after cooling. The hydration capacity was calculated with Equation (3):

$$Hydration \ capacity \ (\%) = \frac{(m_2-m_o)-m_1}{m_1} \times 100 \eqno(3)$$

where m_0 —the weight of the tube, m_1 —the weight of sample taken into analysis, m_2 —the weight of sample which absorbed water.

2.3.2. Water Absorption Capacity

The water absorption capacity was determined in duplicate, according to the method described by Oladiran and Emmambux [39] with modifications. An amount of 2.5 g of the sample was placed in a centrifuge tube with 30 mL of distilled water. The sample was kept in a water bath with continuous stirring at 30 °C for 30 min and after centrifugation at 3500 rpm for 15 min the supernatant was removed and the residue was weighed. The results were calculated by using Equation (4):

Water absorption capacity (%) =
$$\frac{m_1}{m_0} \times 100$$
 (4)

where m_0 —the weight of sample taken into analysis, m_1 —the weight of the sample after supernatant removal.

Appl. Sci. 2021, 11, 11881 4 of 17

2.3.3. Oil Absorption Capacity

Oil absorption capacity was determined in duplicate according to the method described by Elkhalifa and Bernhardt [40] with modifications. An amount of 3 g of sample was placed in a centrifuge tube with 30 mL of sunflower oil. The sample was stirred for 1 min, every 10 min for 30 min. After centrifugation at 3000 rpm for 15 min, the supernatant was decanted and the tubes were allowed to drain for 5 min, then the residue was weighed. The results were calculated by using Equation (5):

Oil absorption capacity (%) =
$$\frac{m_1}{m_0} \times 100$$
 (5)

where m_0 —the weight of sample taken into analysis, m_1 —the weight of the sample after supernatant removal.

2.3.4. Determination of Solubility Index

The method for solubility index determination was adapted according to that presented by Oladiran and Emmambux [39]. The supernatant from the water absorption capacity determination was dried in metal capsules at $100\,^{\circ}\text{C}$ to constant mass and weighed after cooling in the desiccator. The amount of soluble solids expressed as a percentage was defined as the solubility index.

2.3.5. Water Retention Capacity

Water retention capacity was determined in duplicate according to the method described by Zhu et al. [41]. For this purpose, 1 g of sample was placed in a centrifuge tube with 30 mL of distilled water. After 18 h of resting, the sample was centrifuged at 3000 rpm for 20 min and the supernatant was removed. The sample was dried for 2 h at 105 $^{\circ}$ C in a convection oven. The results expressed as an average of two determinations were calculated with Equation (6):

Water retention capacity
$$(g/g) = \frac{m_1 - m_2}{m_2} \times 100$$
 (6)

where m_1 —the weight of the sample before drying, m_2 —the weight of the sample after drying.

2.3.6. Swelling Power

The swelling power was determined according to the method described by Elkhalifa and Bernhardt [40] with modifications: 0.5 g of sample were mixed with 15 mL of distilled water in a weighed centrifuge tube, which was heated in a water bath at 90 °C for 30 min and mixed well to prevent lumps. After cooling to room temperature for 15 min, the sample was centrifuged at 3000 rpm for 25 min. The supernatant was carefully removed and the swollen flour sediment was weighed.

2.3.7. Emulsion Activity and Stability

The emulsifying properties were determined in duplicate according to the method presented by Elkhalifa and Bernhard [40]: 2 g of sample was mixed with 20 mL of distilled water cooled at 4 °C and 20 mL of sunflower oil, in a centrifuge tube. The sample was stirred for 20 min and centrifuged at 4000 rpm for 10 min, then the height of the emulsion layer formed was observed. The emulsion activity was calculated with Equation (7):

Emulsifying activity (%) =
$$\frac{\text{height of emulsion layer}}{\text{height of whole layer}} \times 100$$
 (7)

For emulsion stability evaluation, the emulsion formed was heated in a water bath at 80 °C for 30 min, followed by cooling to room temperature for 20 min. The tube was

Appl. Sci. **2021**, 11, 11881 5 of 17

centrifuged again at 4000 rpm for 10 min. The height of the emulsified layer was measured and the stability of the emulsion was calculated with Equation (8):

Emulsion stability (%) =
$$\frac{\text{height of the emulsion layer after heating}}{\text{height of whole layer}} \times 100$$
 (8)

2.3.8. Bulk Density

For bulk density measurement, 10 g of flour were placed in a 25 mL graduated cylinder. The cylinder was lightly beaten ten times to even out the flour and the final volume of the flour was measured and expressed as g/cm^3 [40].

2.3.9. Foaming Capacity and Stability

Foaming capacity and stability were determined according to the methods described by Elkhalifa and Bernhardt [40]. In a 500 mL beaker, 2 g of sample were transferred with 100 mL of distilled water, and the suspension was mixed with an electric blender, at room temperature, for 1 min. The contents were immediately transferred to a 250 mL graduated cylinder and the volume of foam was measured. The foaming capacity was expressed using the following formula (Equation (9)):

Foaming capacity (%) =
$$\frac{\text{volume after whipping} - \text{volume before whipping}}{\text{volume before whipping}} \times 100$$
 (9)

Foam stability was determined by monitoring the fall in the volume of the foam as a function of time after every 10 min for 1 h and expressed using Equation (10):

Foam stability (%) =
$$\frac{\text{Foam volume after set of time}}{\text{Initial foam volume}} \times 100$$
 (10)

2.4. FT-IR Spectra Collection and Interpretation

FT-IR spectra of sorghum flours were acquired in the range of 650 to 4000 cm⁻¹, from a Thermo Scientific Nicolet iS20 (Waltham, MA, USA) device equipped with and ATR module, at a resolution of 8 cm⁻¹ and with 64 scans. The spectra were analyzed with OMNIC software and the carbohydrates, protein, lipid, and polyphenols structures were identified according to previous studies [42–44].

2.5. Statistical Analysis

Statistically significant differences at 95% confidence level were evaluated by means of three-way ANOVA with Tukey's test, by using XLSTAT for Excel 2021 version (Addinsoft, New York, NY, USA). Principal component analysis (PCA) based on Pearson correlations was employed to evaluate the relationships between the sorghum flour characteristics and to underline similarities or dissimilarities between them.

3. Results

3.1. Proximate Composition

Dry heat treatment, particle size, and sorghum variety significantly influenced (p < 0.05) sorghum flour proximate composition (Table 1). The protein content decreased with particle size decrease, while the increase of treatment temperature led to lower values, red sorghum fractions being richer in protein compared to white variety. Sorghum flour fat content registered proportional increases with particle size reduction, higher amounts being observed in white sorghum compared to the red one. Dry heat treatment produced an increase in fat content as the temperature was higher, depending on the particle size. The ash content showed significant raise as the particle size was lower, slightly higher values being obtained for white sorghum, while dry heat treatment induced a slight decrease of this parameter. Sorghum grain treatment caused flour moisture decrease, while in the case of particle sizes irregular trends were observed. Particle size reduction led to

Appl. Sci. **2021**, 11, 11881 6 of 17

higher carbohydrates content compared to integral flour, except for L particle size, sorghum grain treatment temperature increase determining slightly higher values compared to the control, while the red variety showed higher flour carbohydrates contents than the white one. Sorghum flour fiber content increased until M particle size and then decreased for S samples, grains treatment causing significant increases compared to the control. Red sorghum showed higher fibers content compared to the white one.

Table 1. Effect of dr	y heat treatment on the p	proximate com	position of so	orghum flour fractions.

Treatment	Protein (%)	Fat (%)	Ash (%)	Moisture (%)	Total Dietary Fiber (%)	Carbohydrates (%)	Energetic Value (kcal/100 g)
CW_I	10.35 ± 0.08 ef	3.07 ± 0.03^{ijk}	$1.16 \pm 0.03^{\mathrm{j}}$	11.50 ± 0.01 °	8.35 ± 0.24 cde	$65.56 \pm 0.29^{ i}$	348.01 ± 0.44 hi
CW_L	11.84 ± 0.06 a	3.12 ± 0.01^{ijk}	0.76 ± 0.01 1	11.29 ± 0.01 d	5.38 ± 0.51 k	67.59 ± 0.51 efg	356.59 ± 1.12 ef
CW_M	$8.84\pm0.01~\mathrm{mn}$	3.15 ± 0.03^{ij}	1.15 ± 0.01 ^j	11.20 ± 0.03 e	$5.81 \pm 0.02^{\mathrm{jk}}$	69.86 ± 0.02 abc	$354.74 \pm 0.14 ^{\mathrm{fg}}$
CW_S	10.62 ± 0.19 d	$3.20 \pm 0.03^{ m hi}$	2.26 ± 0.03 a	$10.98 \pm 0.01 ^{\mathrm{g}}$	5.30 ± 0.34 k	67.64 ± 0.43 efg	$352.45 \pm 0.82 \mathrm{g}$
T1W_I	$10.28 \pm 0.07 ^{\mathrm{fg}}$	3.45 ± 0.04 f	1.17 ± 0.01 ^j	8.19 ± 0.04 ^p	$6.18 \pm 0.31^{ ext{ ijk}}$	70.73 ± 0.27 a	367.45 ± 0.65 b
T1W_L	$11.14\pm0.04~^{\rm c}$	$1.62 \pm 0.06^{\text{ n}}$	$0.30 \pm 0.01^{\text{ n}}$	$9.53 \pm 0.01^{\text{ i}}$	$6.49\pm0.58~^{ m hijk}$	70.93 ± 0.62 a	$355.74 \pm 1.08 ^{\mathrm{f}}$
T1W_M	$10.44 \pm 0.05 ^{ m def}$	4.64 ± 0.15 c	$1.54 \pm 0.03 ^{\rm n}$	$9.52 \pm 0.01^{\text{ i}}$	$6.59 \pm 0.10^{ m ghijk}$	$67.26 \pm 0.09 ^{\mathrm{fgh}}$	365.78 ± 0.59 bc
T1W_S	8.68 ± 0.03 no	5.14 ± 0.06 b	2.15 ± 0.01 b	10.01 ± 0.01 ^h	$6.48\pm0.48~^{ m hijk}$	67.53 ± 0.51 efg	364.08 ± 0.87 c
T2W_I	$9.48 \pm 0.06^{\mathrm{jk}}$	3.01 ± 0.02^{k}	$1.37\pm0.01~^{\rm i}$	$8.74 \pm 0.01^{\text{ n}}$	7.14 ± 0.60 efghi	70.25 ± 0.57 ab	360.35 ± 1.29 d
T2W_L	$10.16 \pm 0.03 \mathrm{gh}$	$2.49\pm0.04~^{\rm m}$	0.80 ± 0.07^{1}	$8.73 \pm 0.01^{\text{ n}}$	$6.81\pm0.48~^{ m fghij}$	71.00 ± 0.46 a	360.69 ± 1.05 d
T2W_M	10.08 ± 0.03 hi	4.30 ± 0.03 d	$1.53 \pm 0.03^{\text{ h}}$	$8.96 \pm 0.01^{\text{ k}}$	$7.57 \pm 0.19^{ m \ efgh}$	$67.55 \pm 0.17^{ m \ efg}$	364.40 ± 0.37 c
T2W_S	8.58 ± 0.02 $^{\mathrm{o}}$	6.32 ± 0.03 a	$2.03\pm0.02^{\text{ c}}$	$8.91 \pm 0.01^{\text{ k}}$	$5.42 \pm 0.16^{\text{ k}}$	68.73 ± 0.18 cde	377.00 ± 0.28 a
CR_I	11.40 ± 0.05 b	2.84 ± 0.02^{1}	$1.34 \pm 0.00^{\text{ i}}$	11.08 ± 0.03 f	7.82 ± 0.28 defg	65.51 ± 0.28 i	348.89 ± 0.54 h
CR_L	11.94 ± 0.18 a	2.77 ± 0.04 ¹	0.88 ± 0.04 k	11.15 ± 0.06 e	9.94 ± 0.39 ab	$63.32 \pm 0.54^{\text{ j}}$	$345.84 \pm 0.85^{\text{ i}}$
CR_M	$9.66 \pm 0.18^{\mathrm{j}}$	3.10 ± 0.03^{ijk}	1.75 ± 0.03 f	11.80 ± 0.03 b	$7.69 \pm 0.39^{\text{ defgh}}$	66.00 ± 0.36 hi	$345.91 \pm 0.80^{\text{ i}}$
CR_S	10.58 ± 0.13 d	3.28 ± 0.04 gh	2.30 ± 0.03 a	12.01 ± 0.05 a	$6.49\pm0.31^{ m \ hijk}$	$65.33 \pm 0.31^{\mathrm{\ i}}$	$346.16 \pm 0.60^{\ i}$
T1R_I	10.53 ± 0.05 de	2.73 ± 0.16^{1}	$1.39 \pm 0.04^{\mathrm{\ i}}$	$8.02 \pm 0.03 ^{ ext{ q}}$	9.41 ± 0.08 bc	67.92 ± 0.06 defg	357.21 ± 0.86 ef
T1R_L	11.48 ± 0.03 b	$1.16\pm0.04~^{\rm o}$	0.66 ± 0.01 ^m	8.80 ± 0.02 lm	10.75 ± 0.95 a	$67.14 \pm 0.98 ^{ m gh}$	346.43 ± 1.85 hi
T1R_M	9.29 ± 0.03^{1}	$3.40\pm0.03~^{\mathrm{fg}}$	1.52 ± 0.01 h	$8.75 \pm 0.01 \mathrm{mn}$	8.38 ± 0.40 ^{cde}	68.65 ± 0.39 cde	359.13 ± 0.88 de
T1R_S	8.51 ± 0.03 $^{\mathrm{o}}$	4.04 ± 0.06 e	$1.91 \pm 0.03^{\text{ e}}$	$8.94 \pm 0.01^{\text{ k}}$	8.02 ± 0.01 def	$68.58 \pm 0.10^{ m cdef}$	360.75 ± 0.07 d
T2R_I	9.35 ± 0.00 kl	3.04 ± 0.04 jk	1.54 ± 0.01 h	8.67 ± 0.01 $^{\mathrm{o}}$	$7.64 \pm 0.37^{ m \ defgh}$	69.76 ± 0.37 abc	359.07 ± 0.66 de
T2R_L	$9.93 \pm 0.05^{\ i}$	$1.62 \pm 0.03^{\ n}$	0.93 ± 0.01 k	8.82 ± 0.02 ¹	8.11 ± 0.68 cdef	70.58 ± 0.67 a	$352.88 \pm 1.30 ^{\mathrm{g}}$
T2R_M	9.21 ± 0.05^{1}	$3.40\pm0.03~^{\mathrm{fg}}$	$1.64\pm0.01~^{\rm g}$	$9.07 \pm 0.01^{\ \mathrm{j}}$	$7.64\pm0.26~^{ m defgh}$	69.03 ± 0.29 bcd	358.90 ± 0.48 de
T2R_S	8.93 ± 0.03 m	4.13 ± 0.03 e	1.97 ± 0.01 d	8.96 ± 0.01 k	8.95 ± 0.51 bcd	67.05 ± 0.52 gh	359.02 ± 0.94 de

CW—Control white sorghum, CR—control red sorghum, T1—dry heat treatment at 121 °C, T2—dry heat treatment at 140 °C, L/M/S—particle sizes, I—integral. Each experiment was carried out in duplicate and data were reported as means \pm standard deviation (SD). Means in the same column with different letters are significantly different (p < 0.05).

Dry heat treatment caused an increase in energetic value compared to the control and with temperature raise, the values for white and red varieties being close to each other. An increasing trend for the energetic values was observed in treated samples with particle size reduction, while for CW the opposite trend occurred.

3.2. Functional Properties

Sorghum grains dry heat treatment, milling and variety showed significant variations (p < 0.05) in flours functional properties. The water absorption capacity of flours generally decreased as the particle size was lower, except for CR, CW, and T1W which showed the highest values for the M fraction (Table 2). Sorghum grains dry heat treatment led to an increased water absorption capacity. Flours oil absorption capacity raise was proportional with particle size reduction, while red sorghum variety showed slightly higher values compared to the white one, and dry heat treatment produced irregular small changes depending on the particle size. Water retention capacity registered the lowest values in the case of M particle size, except for the T2R sample where it was the greatest, close values being observed between the two varieties, sorghum treatment determining a decrease of this parameter with temperature increase. Flours hydration capacity decreased with particle size reduction, except for treated white sorghum with M particle size and red sorghum with L particle size samples.

Appl. Sci. **2021**, 11, 11881 7 of 17

Table 2. Effect of dry heat treatment on the water absorption, oil absorption capacity, water retention capacity, hydration capacity, swelling, and solubility index of sorghum fractions.

Treatment	Water Absorption Capacity (%)	Oil Absorption Capacity (%)	Water Retention Capacity (g/g)	Hydration Capacity (%)	Swelling Power (g/g)	Solubility Index (%)
CW_I	$208.80 \pm 1.13~^{\text{defghi}}$	$162.05 \pm 0.25~\mathrm{g}$	1.29 ± 0.04 ab	92.41 ± 0.55 fgh	3.35 ± 0.01 kl	0.10 ± 0.00 ab
CW_L	$202.00 \pm 1.41^{\text{ hij}}$	$152.36 \pm 0.76^{\ ij}$	1.31 ± 0.11 ab	$98.11 \pm 1.26^{\text{ e}}$	$3.51 \pm 0.01^{\text{ h}}$	$0.04 \pm 0.00 ^{ m fgh}$
CW_M	$208.60\pm0.28~^{\rm defghi}$	166.71 ± 0.54 e	$1.06\pm0.03~{ m defg}$	$97.80 \pm 1.98^{\text{ e}}$	3.98 ± 0.01 d	0.12 ± 0.01 a
CW_S	$207.40\pm0.14~^{\mathrm{efghij}}$	171.33 ± 0.61 bc	1.32 ± 0.02 ab	$91.30 \pm 2.12 ^{ m ghi}$	4.31 ± 0.01 a	0.08 ± 0.00 bc
T1W_I	$201.00 \pm 0.54^{ m hij}$	163.45 ± 0.00 f	1.30 ± 0.11 bcd	$94.60 \pm 0.30^{\text{ f}}$	3.38 ± 0.07 jk	0.08 ± 0.00 bc
T1W_L	$209.10 \pm 1.27 ^{ m defgh}$	151.95 ± 0.41 jk	1.21 ± 0.03 abcd	94.58 ± 0.01 f	3.28 ± 0.00 m	$0.02 \pm 0.00 ^{ m h}$
T1W_M	216.50 ± 0.71 bcd	176.99 ± 0.47 a	0.84 ± 0.01 h	99.60 ± 0.57 cde	$3.82 \pm 0.00^{\text{ e}}$	$0.03 \pm 0.01 \mathrm{gh}$
T1W_S	$200.50 \pm 0.71^{\ ij}$	170.99 ± 0.15 bc	0.85 ± 0.05 h	$89.00 \pm 0.28^{\ i}$	4.02 ± 0.00 ^{cd}	$0.04\pm0.00~^{ m fgh}$
T2W_I	199.00 ± 1.98 jk	166.38 ± 0.08 e	$1.05\pm0.01^{ m \ defg}$	99.40 ± 1.13 ^{cde}	4.04 ± 0.00 ^c	$0.03 \pm 0.00 \mathrm{gh}$
T2W_L	221.60 ± 1.41 ^{cde}	151.01 ± 0.49 k	$0.89 \pm 0.13 ^{\mathrm{fgh}}$	101.29 ± 0.43 bcd	$3.42 \pm 0.00^{\text{ ij}}$	$0.02 \pm 0.00 ^{\rm h}$
T2W_M	219.40 ± 3.11 abc	166.53 ± 0.93 de	$0.77 \pm 0.00^{\text{ h}}$	102.29 ± 0.42 b	$4.21 \pm 0.00^{\ \mathrm{b}}$	$0.03 \pm 0.00 \mathrm{gh}$
T2W_S	209.20 ± 2.26 cdef	170.90 ± 0.61 bc	0.81 ± 0.04 ^h	$89.91 \pm 0.30 \mathrm{hi}$	4.35 ± 0.00 a	$0.05\pm0.03~\mathrm{efg}$
CR_I	$208.78 \pm 1.72 ^{ m defghi}$	163.97 ± 0.61 f	$1.27\pm0.14~^{ m abc}$	$93.30 \pm 0.42 \mathrm{f}\mathrm{g}$	3.32 ± 0.00 lm	0.11 ± 0.00 a
CR_L	$199.50 \pm 0.70^{\mathrm{jk}}$	$160.11 \pm 0.83^{\text{ h}}$	1.39 ± 0.01 a	98.74 ± 0.88 de	3.32 ± 0.00 lm	0.08 ± 0.00 bc
CR_M	$209.55 \pm 0.78 ^{ m defg}$	171.66 ± 0.60 b	1.09 ± 0.01 cde	$97.50 \pm 0.99^{\text{ e}}$	$3.63 \pm 0.00 \mathrm{g}$	0.05 ± 0.00 efg
CR_S	$209.30 \pm 0.14 ^{ m defgh}$	170.00 ± 0.82 ^c	1.30 ± 0.02 ab	$91.65 \pm 1.48 ^{ m gh}$	$3.70\pm0.01~^{\mathrm{f}}$	0.07 ± 0.00 cde
T1R_I	$201.99 \pm 1.68 ^{ m ghij}$	168.05 ± 0.08 d	1.21 ± 0.11 abcd	$93.04 \pm 3.08 ^{\mathrm{fg}}$	$3.43\pm0.00^{\mathrm{\;i}}$	0.08 ± 0.01 bcd
T1R_L	226.80 ± 0.57 a	160.00 ± 0.48 h	$0.87 \pm 0.08 \mathrm{gh}$	107.71 ± 1.00 a	$3.37 \pm 0.00^{\ k}$	$0.05 \pm 0.05 \mathrm{defg}$
T1R_M	210.08 ± 0.96 defg	168.16 ± 0.71 ^d	$0.90 \pm 0.03 ^{ m efgh}$	101.50 ± 0.42 bc	$3.60 \pm 0.01 \mathrm{g}$	$0.03 \pm 0.00 \mathrm{gh}$
T1R_S	191.10 ± 0.99 k	170.99 ± 0.61 bc	$0.88 \pm 0.01 ^{ m fgh}$	91.21 ± 0.83 ghi	$3.68 \pm 0.01 ^{ ext{ f}}$	$0.04 \pm 0.00 ^{ m fgh}$
T2R_I	$203.39 \pm 3.12 ^{ m fghij}$	$163.50 \pm 0.70^{\text{ f}}$	$1.07\pm0.02~\mathrm{def}$	105.50 ± 1.27 a	3.77 ± 0.08 e	0.06 ± 0.02 ^{cdef}
T2R_L	224.76 ± 5.71 ab	153.65 ± 0.66 i	$0.87 \pm 0.06 ^{\mathrm{gh}}$	107.60 ± 0.28 a	3.32 ± 0.00 lm	$0.02 \pm 0.00^{\text{ h}}$
T2R_M	210.08 ± 0.57 abc	167.27 ± 0.87 de	1.31 ± 0.26 $^{\mathrm{ab}}$	107.10 \pm 0.15 $^{\mathrm{a}}$	3.42 ± 0.00^{ij}	0.02 ± 0.01 ^h
T2R_S	$210.08\pm1.41~^{\mathrm{cde}}$	$170.09\pm0.94^{\text{ c}}$	0.89 ± 0.01 fgh	$93.01 \pm 0.59 ^{\mathrm{fg}}$	$3.79 \pm 0.00^{\text{ e}}$	$0.05\pm0.01~\mathrm{efg}$

CW—Control white sorghum, CR—control red sorghum, T1—dry heat treatment at 121 °C, T2—dry heat treatment at 140 °C, L/M/S—particle sizes, I—integral. Each experiment was carried out in duplicate and data were reported as means \pm standard deviation (SD). Means in the same column with different letters are significantly different (p < 0.05).

Dry heat treatment induced slight increases in flour hydration capacity in almost all cases, while red sorghum flours presented higher values compared to the white variety. A proportional raise of swelling power was obtained with particle size decrease, while white sorghum flours showed higher values compared to the red ones. Solubility index decreased with particle size reduction for almost all the tested samples (except for CW_M), and dry heat treatment led to the slight decrease of these parameter's values. Flour bulk density was influenced irregularly by particle size, the lowest values being observed for M particle sizes (Table 3). A similar irregular trend was determined by dry heat treatment, while the values between the two sorghum varieties were close.

The emulsifying activity recorded significant decreases (p < 0.05) with particle size reduction, except for L for both red and white varieties, while dry heat treatment induced irregular changes. Emulsion stability recorded higher values for L and S compared to M particle size for both sorghum varieties. The increase in temperature determined the increase of emulsion stability in all fractions of white sorghum, excepting T2W_S, while for the red variety the values followed an irregular trend.

Appl. Sci. 2021, 11, 11881 8 of 17

Table 3. Effect of dry heat treatment on the bulk density and emulsifying properties of sorghum flour fractions.

	Bulk	Emulsifyin	g Properties	Foaming Stability (%)								
Treatment	Density (g/cm ³)	Emulsifying Activity (%)			20 min	30 min	40 min	50 min	60 min			
CW_I	$0.71 \pm 0.01^{\ ij}$	$47.50 \pm 0.72 ^{\mathrm{fg}}$	$60.50 \pm 0.72 ^{\mathrm{fg}}$	115.00 ± 0.00 ab	110.00 ± 0.00 bc	110.00 ± 0.00 bc	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 bc			
CW_L	$0.83 \pm 0.00 ^{\mathrm{d}}$	56.50 ± 0.72 a	$66.50\pm0.72~^{\mathrm{a}}$	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	$110.00\pm0.00~\mathrm{ab}$			
CW_M	$0.70 \pm 0.00^{\mathrm{jk}}$	$45.50 \pm 0.72 \mathrm{ghi}$	$54.50 \pm 0.72^{\mathrm{j}}$	120.00 ± 0.00 a	$120.00\pm0.00~\textrm{a}$	$120.00 \pm 0.00~^{a}$	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 a			
CW_S	$0.72 \pm 0.00 \mathrm{ghi}$	$41.50 \pm 0.72^{\mathrm{jk}}$	62.50 ± 0.72 de	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	105.00 ± 0.00 ^c	105.00 ± 0.00 bc			
T1W_I	$0.70 \pm 0.00^{\mathrm{jk}}$	50.00 ± 1.41 de	61.50 ± 0.72 ef	115.00 ± 0.00 ab	110.00 ± 0.00 bc	110.00 ± 0.00 ab						
T1W_L	$0.88 \pm 0.00^{\ \mathrm{b}}$	48.50 ± 0.72 ef	$59.50 \pm 0.72 \mathrm{g}$	-	-	-	-	-	-			
T1W_M	0.62 ± 0.00 ^m	$41.50 \pm 0.72^{\mathrm{jk}}$	61.50 ± 0.72 ef	115.00 ± 0.00 ab	110.00 ± 0.00 bc	110.00 ± 0.00 ab						
T1W_S	$0.73 \pm 0.00 \mathrm{gh}$	$41.50 \pm 0.72^{\mathrm{jk}}$	62.50 ± 0.72 de	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	115.00 ± 0.00 ab	115.00 ± 0.00 a			
T2W_I	0.64 ± 0.01 1	47.50 ± 0.72 fg	63.50 ± 0.72 cd	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	105.00 ± 0.00 bc			
T2W_L	0.89 ± 0.01 $^{\mathrm{ab}}$	$54.00 \pm 1.41^{\ \mathrm{b}}$	64.50 ± 0.72 bc	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 bc			
T2W_M	$0.62 \pm 0.00 \text{ m}$	48.50 ± 0.72 ef	65.50 ± 0.72 $^{ m ab}$	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	110.00 ± 0.00 bc	-			
T2W_S	$0.73 \pm 0.00 \mathrm{gh}$	38.50 ± 0.72^{1}	$56.50\pm0.72^{ m hi}$	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	115.00 ± 0.00 a			
CR_I	$0.72\pm0.01~^{\mathrm{fg}}$	51.50 ± 0.72 cd	63.50 ± 0.72 cd	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 ab			
CR_L	0.90 ± 0.00 a	53.50 ± 0.72 bc	$62.50 \pm 0.72 \mathrm{de}$	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	110.00 ± 0.00 bc	105.00 ± 0.00 c	105.00 ± 0.00 bc			
CR_M	$0.69 \pm 0.00^{\text{ k}}$	$44.00\pm1.41^{\rm \ i}$	55.50 ± 0.72^{ij}	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 $^{ m ab}$	105.00 ± 0.00 bc			
CR_S	$0.69 \pm 0.01^{\text{ k}}$	$43.50 \pm 0.72^{\ ext{ij}}$	62.50 ± 0.72 de	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	110.00 ± 0.00 ab			
T1R_I	0.72 ± 0.01 hij	46.50 ± 0.72 fgh	$59.50 \pm 0.72 \mathrm{g}$	115.00 ± 0.00 $^{\mathrm{ab}}$	115.00 ± 0.00 ab	110.00 ± 0.00 $^{\mathrm{ab}}$						
T1R_L	$0.86\pm0.00~^{\rm c}$	55.50 ± 0.72 ab	62.50 ± 0.72 de	-	-	-	-	-	-			
T1R_M	0.65 ± 0.00^{1}	46.50 ± 0.72 fgh	$61.50 \pm 0.72 \mathrm{efg}$	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	110.00 ± 0.00 bc	110.00 ± 0.00 bc	-			
T1R_S	$0.75 \pm 0.00 \mathrm{ef}$	$43.50 \pm 0.72^{\ ij}$	61.50 ± 0.72 ef	120.00 ± 0.00 a	120.00 ± 0.00 a	120.00 ± 0.00 a	115.00 ± 0.00 ab	115.00 ± 0.00 $^{ m ab}$	115.00 ± 0.00 a			
T2R_I	0.65 ± 0.00^{1}	50.00 ± 1.41 de	$57.50 \pm 0.72^{\text{ h}}$	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	100.00 ± 0.00 ^c			
T2R_L	$0.83 \pm 0.00^{\text{ d}}$	55.00 ± 1.41 ab	64.00 ± 1.41 bcd	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 ^c	105.00 ± 0.00 bc			
T2R_M	0.75 ± 0.01 ef	$44.50\pm0.72~^{ m hi}$	$60.50\pm0.72~\mathrm{fg}$	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	-	-			
T2R_S	0.76 ± 0.00 e	$40.50\pm0.72~^{\mathrm{kl}}$	61.50 ± 0.72 ef	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	115.00 ± 0.00 ab	$105.00\pm0.00~^{\rm c}$	105.00 ± 0.00 bc			

CW—Control white sorghum, CR—control red sorghum, T1—dry heat treatment at 121 $^{\circ}$ C, T2—dry heat treatment at 140 $^{\circ}$ C, L/M/S—particle sizes, I—integral. Each experiment was carried out in duplicate and data were reported as means \pm standard deviation (SD). Means in the same column with different letters are significantly different (p < 0.05).

Appl. Sci. 2021, 11, 11881 9 of 17

Flour foaming capacity was affected by particle size, dry heat treatment, and sorghum variety (Figure 1). Particle size reduction led to the increase of foaming capacity, the differences between varieties being not noticeable. On the other hand, dry heat treatment caused lower foaming capacity values compared to the controls and with temperature increase, excepting T2R_S.

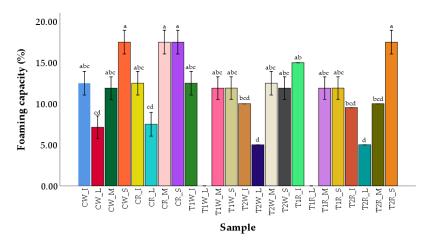


Figure 1. Effect of dry heat treatment on sorghum flour fractions foaming capacity: CW—Control white sorghum, CR—control red sorghum, T1—dry heat treatment at 121 $^{\circ}$ C, T2—dry heat treatment at 140 $^{\circ}$ C, L/M/S—particle sizes, I—integral. Means with distinct letters are significantly different (p < 0.05).

The variation of red and white sorghum flours foaming stability with dry heat treatment temperature increase and particle size decrease in time is presented in Table 3. Foaming stability registered an increase with particle size decrease, except for CW where an irregular trend was observed. Sorghum grains dry heat treatment caused an irregular variation of foaming stability, the lowest values being observed for T2W_L and T2R_L, the samples treated at 121 °C (T1W_L and T1R_L not presenting any foaming capacity). As expected, foaming stability decreased in time for all the studied samples.

3.3. Molecular Characteristics

FT-IR spectra of red and white untreated and treated sorghum flours with different particle sizes are shown in Figure 2. Many peaks can be observed on the FT-IR spectra that could be associated with the molecular bindings of sorghum chemical compounds such as starch, proteins, and polyphenols. Particle size reduction, sorghum variety, and dry heat treatment led to changes in the intensities of absorption band and the appearance of peaks in some regions. There can be observed two prominent peaks in the region 1700–1600 cm⁻¹ (at 1649 cm⁻¹) and in the region 1060–960 cm⁻¹ (997 cm⁻¹), the first one being ascribed to Amide I expression of proteins, while the second band to carbohydrates fingerprint [45]. The peaks found in 3800–3600 cm⁻¹ may be attributed to the O-H groups of phenols intermolecular bonded [36], while the peak found at about 3296 cm⁻¹ was given by the O-H stretching vibration [46].

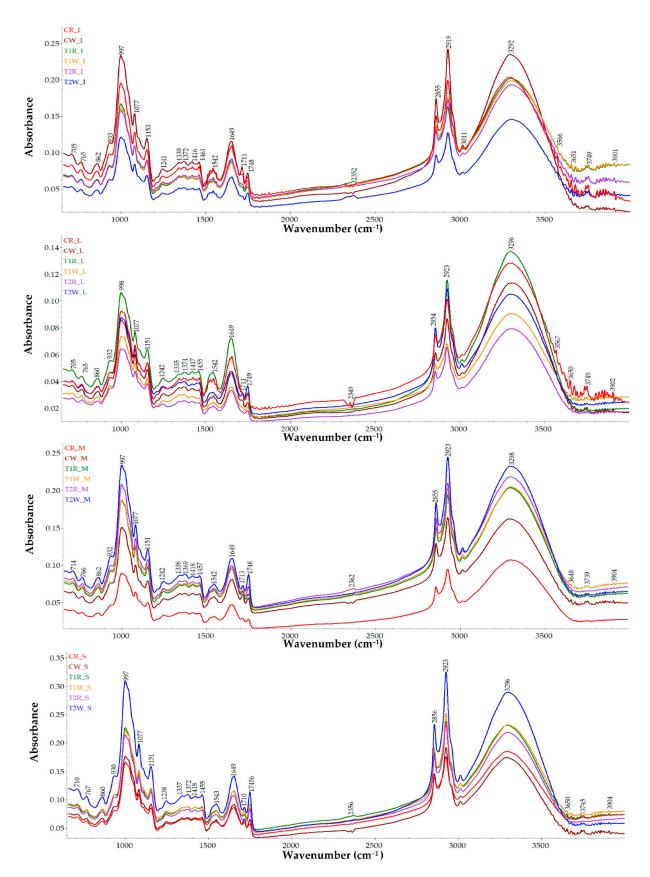


Figure 2. FT-IR spectra of untreated (C) and treated (T1, T2) red (R) and white (W) sorghum flours at different particle sizes: integral (I), large (L), medium (M), and small (S).

The absorption bands found at about 2923 and 2352 cm⁻¹ could be due to the C-H stretching vibration and could suggest the presence of the alkane group and/or cis-olefinic group [2]. Interesting changes of FT-IR spectra were observed in the 2352 cm⁻¹ region, in CR L, CW S, CW M, T2R M, and integral flours samples which showed higher peak absorbances compared to other samples (Figure 3). The absorption bands found at about 1151 and 1077 cm⁻¹ could be attributed to the fiber fractions such as small hemicellulose and cellulose [45]. On the other hand, 1154 and 1416 could be due to the P-H, P-H banding, phosphine, and phosphoric acid which may be associated with the presence of phytic acid [43]. In the integral flours, the reduction of absorption intensity in 1154 and 1416 cm $^{-1}$ was observed with the increase of sorghum grain dry heat treatment temperature. The peaks observed in 750–880 cm⁻¹ region and 1150–1500 cm⁻¹ zone could be associated with aromatic rings deformation, revealing the presence of phenols, with stretching given by them and C-C, C-H, O-H deformations suggesting the presence of phenolic acids and flavonoids [42]. The decrease of particle size led to higher absorbances in these regions, while white sorghum exhibited higher absorbances compared to the red one, except for L particle size. Dry heat treatment showed differential contribution in absorbances intensities, higher values compared to the controls being observed in the case of L, M, and S particle sizes. For the L particle size sample, the highest absorption intensities were observed for T1R_L, while for M and S particle sizes T2W samples exhibited the highest absorbances compared to the other studied samples.

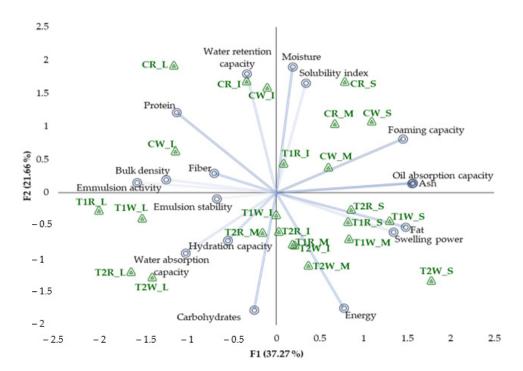


Figure 3. Principal Component Analysis (PCA) bi-plot: CW—Control white sorghum, CR—control red sorghum, T1—dry heat treatment at 121 °C, T2—dry heat treatment at 140 °C, L/M/S—particle sizes, I—integral.

3.4. Relations between Variables

The correlations between variables are presented in Table 4. Foaming capacity was positively correlated with the fat (r = 0.52, p < 0.05) and ash contents (r = 0.78, p < 0.05) and negatively with carbohydrates (r = -0.37, p < 0.05). Oil absorption capacity was correlated at p < 0.05 significance level with fat (r = 0.67), ash (r = 0.84), protein (r = -0.50) and foaming capacity (r = 0.72). Moderate correlations were observed between hydration capacity and ash (r = -0.46, p < 0.05), fat contents (r = -0.48, p < 0.05) and foaming capacity (r = 0.44), fat (r = 0.44), fat power was positively correlated with foaming capacity (r = 0.44), fat

Appl. Sci. 2021, 11, 11881 12 of 17

(r=0.69), ash (r=0.66) and oil absorption capacity (r=0.60) and negatively with protein content (r=-0.51) and fibers (r=-0.48), significant at p<0.05 level. Moderate correlations were obtained between solubility index and flours moisture content (r=0.48, p<0.05), carbohydrates (r=-0.36, p<0.05) and water retention capacity (r=0.45, p<0.05). Bulk density was positively correlated with the protein content (r=0.45) and negatively with fat (r=-0.50), ash (r=-0.59) and oil absorption capacity (r=-0.73), significant at p<0.05. Similar, but stronger correlations were obtained between emulsifying activity and protein content (r=0.64), fat (r=-0.77), ash (r=-0.80), oil absorption capacity (r=-0.84) and hydration capacity (r=0.46, p<0.05). Emulsion stability was moderately correlated with the protein content of flours (r=0.46, p<0.05). Significant negative (p<0.05) correlations between carbohydrates and moisture (r=-0.63) and carbohydrates and fibers (r=-0.40) were obtained, while fat and ash contents were positively correlated (r=0.69). The hydration capacity, swelling power and emulsifying activity were moderate correlated with fiber content (p<0.05).

Variables	FC	Protein	Fat	Ash	Moisture	Fiber	Carbohydrates	Energy	WAC	OAC	WRC	HC	SP	SI	BD	EA	ES
FC	1.00	-0.34	0.52	0.78	0.27	-0.16	-0.37	0.04	-0.26	0.72	0.11	-0.50	0.44	0.35	-0.63	-0.64	-0.20
Protein		1.00	-0.57	-0.57	0.33	0.19	-0.38	-0.53	0.09	-0.50	0.53	0.13	-0.51	0.18	0.45	0.64	0.46
Fat			1.00	0.69	-0.04	-0.40	-0.12	0.70	-0.23	0.67	-0.35	-0.48	0.69	-0.04	-0.50	-0.72	-0.17
Ash				1.00	0.12	-0.23	-0.26	0.23	-0.18	0.84	-0.14	-0.46	0.66	0.08	-0.59	-0.80	-0.17
Moistur	re				1.00	-0.23	-0.63	-0.62	-0.14	0.05	0.52	-0.33	0.00	0.48	0.05	-0.04	-0.08
Fiber						1.00	-0.40	-0.45	0.18	-0.07	-0.07	0.34	-0.48	0.00	0.22	0.31	0.08
Carbohydr	rates						1.00	0.54	0.08	-0.31	-0.32	0.27	0.06	-0.38	0.01	0.09	-0.11
Energy								1.00	-0.09	0.24	-0.52	-0.13	0.49	-0.36	-0.31	-0.38	-0.06
WAC									1.00	-0.15	-0.32	0.56	-0.09	-0.24	0.12	0.14	0.08
OAC										1.00	-0.20	-0.37	0.60	0.17	-0.73	-0.84	-0.36
WRC											1.00	-0.18	-0.37	0.45	0.18	0.19	0.01
HC												1.00	-0.29	-0.38	0.12	0.55	0.14
SP													1.00	-0.01	-0.55	-0.64	-0.16
SI														1.00	-0.15	-0.03	-0.33
BD															1.00	0.54	0.25
EA																1.00	0.46
ES																	1.00

Table 4. Pearson's correlation coefficients.

Values in bold are significant at p < 0.05, FC—foaming capacity, WAC—water absorption capacity, OAC—oil absorption capacity, WRC—water retention capacity, HC—hydration capacity, SP—swelling power, SI—solubility index, BD—bulk density, EA—emulsifying activity, ES—emulsion stability.

Similarities and oppositions between variables were underlined by means of Principal Component Analysis (Figure 3), 58.93% of the total variance being explained. The first component (PC1) explained 37.27% of data variation, while the second one (PC2) explained 21.66% of the variance.

Emulsifying activity, bulk density, fiber content swelling power, fat content, oil absorption capacity, ash content, and foaming capacity were associated with PC1, while moisture content, solubility index, water retention capacity, carbohydrates content, and energetic value were associated with PC2. Emulsion stability, fiber content and hydration capacity position close to the origin underlined their smaller contribution to the data variation.

Control samples with different particle sizes were positioned in the upper part of the graphic and were associated with water retention capacity, moisture content, solubility index, and protein content, while treated sorghum flours with different particle sizes were placed on the lower side. Treated samples with L particle size were grouped and were associated with water absorption capacity, hydration capacity, and emulsifying activity. On the other hand, treated samples with M and S particle sizes were associated with energetic value, swelling power, fat and ash contents, foaming capacity, solubility index and oil absorption capacity.

4. Discussion

The nutritional and functional characteristics of food products are affected by structure-property relationships [24]. Modifications of chemical components proportions and functional properties determined by dry heat treatment and milling have been reported for cereals [24,25]. Protein content decrease with particle size decrease (Table 1) could be due to the localization of nutrients in the grain, being known that aleurone layer in the pericarp and the peripheral endosperm tissue are formed of cells with high amounts of proteins [47].

Fat and ash contents increases and carbohydrates decrease with particle size reduction agreed with the results reported by Alvarenga et al. [47]. In the case of a constant milling process, differences in dissociation between constituent parts of the sorghum grains are determined by the ability to remove the aleurone layer from the peripheral endosperm, the intercellular adherence at the interface of aleurone-endosperm layers being determined by the degree of bonding of arabinoxylan chains [48]. Another important aspect is led to the distribution of endosperm proteins such as kafirins and glutelins [48]. Our results showed that dry heat treatment induced the decrease of moisture and protein, and raised fat, carbohydrates and energetic value. Protein content decrease could be possibly attributed to the damage of amino acids as a result of heat [49]. He et al. [50] also reported increased metabolizable energy and net energy values induced by sorghum heat-related processing. The differences among red and white sorghum chemical composition were in line with those reported by Vargas-Solórzano et al. [48]. The significant (p < 0.05) correlations between, carbohydrates and ash, and between fat and ash contents were in agreement with those reported by Queiroz et al. [51] for sorghum chemical constituents. The results obtained for sorghum fractions fiber content were in line with those reported in the literature [52], the genotype and extraction rate playing a decisive role in fiber variation [26]. The dietary fiber of sorghum grains is mainly composed of cellulose and pentosan [53].

The water absorption capacity of flours decreased as the particle size was lower (Table 2) probably due to the loss of fiber which has great potential to hold water [54] and/or to the different chemical compounds of milling fractions and/or to the particle size distribution and its morphology [55]. For the acceptable food texture of baked goods, higher values for water absorption capacity are recommended [56]. Dry heat treatment caused the increase of water absorption capacity, similar to the trend reported by Adebowale et al. [57] for red sorghum starch treated by annealing and heat moisture treatment, probably due to the starch amylose and amylopectin chains reorganizations. Fat enhances the flavor retention and mouth feel of food products which means that oil retention capacity is an important quality characteristic to govern the ability of flour to physically entrap fat content [25]. Oil absorption capacity increase with particle size decrease could be possibly explained by the presence of lignin, its structure and surface characteristics, overall charge density, thickness, hydrophobic character, and particle size [58]. Grains milling may affect the absorption characteristics due to the increase in surface area. The differences between white and red sorghum could be due to the concentration of hydrophobic amino acids as lipid binding is determined by their concentration [54]. Water retention capacity showed the lowest values for M particle size samples (except for T2R). The hydrophilic compounds found in milling fractions such as polysaccharides present high water retention capacity, while the polar amino acid residues of proteins exert affinity for water molecules, enhancing thus the water binding capacity [55]. Dry heat treatment of sorghum grains caused the decrease of water retention capacity which may be explained by the changes induced by heat such as protein denaturation, starch gelatinization, and swelling of the crude fiber [59], a statement supported also by the correlations (p < 0.05) obtained between water retention capacity with protein and moisture content (Table 4). Flour swelling power increases with particle size reduction, probably as a result of starch damage during milling [60]. Swelling power is related to the intermolecular non-covalent linkages of starch, the degree of swelling being influenced by the molecular weight distribution, amylose-amylopectin ratio, and chain length [54]. Protein solubility decrease caused by dry heat treatment of sorghum grains could be responsible for solubility index decrease. Protein solubility is influenced by the intrinsic factors such as hydrophilic and hydrophobic properties of protein molecules, their dimension and charge, and the interaction with other grain components, and also by external factors such temperature [61]. Bulk density as an indicator for flour heaviness was influenced in an irregular way (Table 3) by particle size, similar to the results presented by Cairano et al. [60]. The presence of fat which could play a binder role in agglomeration of flour particles and the milling process conditions can affect flour granulometry and, thus, bulk density [60], a fact also supported by the significant correlation (p < 0.05) between

Appl. Sci. 2021, 11, 11881 14 of 17

bulk density and fat content (Table 4). In the case of sorghum flour samples with low lipid content (T1W_L, T2W_L, T1R_L, and T2R_L), the increase in bulk density was observed, and therefore using these flours may offer a significant advantage in terms of baked product volume [62]. The emulsion activity and stability values were in line with those reported by Siroha et al. [63] for millet flours and decreased with particle size reduction which may be related to the variations of the interfacial tension and surface hydrophobicity. Flour foaming capacity is determined by protein molecules structures and carbohydrates content [64], a fact confirmed also by the significant correlation (p < 0.05) obtained in this study. Particle size reduction led to the increase of foaming capacity and stability (Figure 1 and Table 3) probably due to the increase in proteins ability to form an elastic, flexible, and cohesive interfacial film that can catch and maintain air for enough time to slow down the coalescence rate [54]. Sorghum grains heat treatment induced the reduction of flour foaming capacity may be due to protein denaturation.

Regarding the molecular characteristics (Figure 3), the decrease of particle size determined proportional absorbances increases, dry heat treatment presenting differential contribution in absorbances intensities by inducing higher values compared to the controls in the case of L, M, and S particle sizes. The differences found at 3011 and 3292 cm⁻¹ may be attributed to the changes in the activity of N-H of primary and secondary amines and/or O-H of either carboxylic acid, alcohols, or starch [36]. These modifications of the amine groups in 3292 cm⁻¹ may also be assigned to their reactions with reducing sugars and α -dicarbonyls, resulting in pyrazinium radical cation and leading to the formation of brown pigments in the heat-treated sorghum flour [36]. The changes of FT-IR spectra intensities for CR L, CW S, CW M, T2R M samples at about 2352 cm⁻¹ could be possibly attributed to the -NH₃⁺ changes in amines or hydrohallides and -PH in the phosphine functional groups [36]. Furthermore, Maillard reactions could determine the raise of unsaturated carbonyl compounds, the disintegration of amino acids to aldehydes, and their condensation with carbohydrates parts, furfurals, and other compounds to form chromophores and off-flavors and could be possibly led to the changes in 2300–2400 cm⁻¹ region [36]. The changes in absorbances values at about 1151 and 1077 cm⁻¹ could be related to the distribution of fiber fractions in the flours with different particle sizes [45]. The reduction of phytic acid content after sorghum grains dry heat treatment was observed by the decrease in absorbances at 1154 and 1416 [43], confirming thus the applicability of this kind of treatment for antinutrients reduction. The peaks found in the region 1200 to 1900 cm⁻¹ are associated with different functional groups such as amides, amino acids, -C=O in aldehydes, C-O in esters, CO in anhydrides, =O in lactones, t-butyl groups, N-O pyridine groups, esters, lactones [36]. The production of chromophores and condensation reactions occurrence, such as reactions of furfurals or dehydro-reductones during Maillard reaction result in the formation of unsaturated brown nitrogenous polymers named melanoidins, depending on the heat treatment condition [36,65]. The changes observed in region 900–1100 cm⁻¹ could be determined by the angular C-H linkage deformation in the sorghum flour, skeletal vibration of 1-4 glycosidic bonds (C-O-C), development of new groups, and stretching vibration of the C-O bond in the esters formed between the non-starch component such as -COOH in the protein and starch molecules [36].

5. Conclusions

Sorghum grain is an important alternative to conventional corps and its functionality and nutritional value can be modified by dry heat treatment, depending on the particle size. Dry heat treatment applied to white and red sorghum grains resulted in higher fat and fiber and lower protein, moisture, and ash contents. Particle size reduction caused protein content decrease and fat contents increase. The results of this study showed that red sorghum is richer in protein, ash, and fibers and less abundant in fat compared to the white variety. Dry heat treatment led to higher flour water absorption and lower water retention capacity, solubility index, and foaming capacity as the temperature increased. Particle size decrease induced the raise of flour oil absorption capacity, swelling power,

Appl. Sci. 2021, 11, 11881 15 of 17

and foaming capacity, while flour solubility index, emulsifying activity decreased. The molecular characteristics of sorghum flours showed significant differences among varieties, particle sizes, and dry heat treatment temperatures. Grains heat treatment led to lower intensities of peaks characteristics for phytic acid, suggesting the utility of this process in antinutrients diminishing. These results evidenced the opportunity to use dry heat treatment to enhance the nutritional and functional profiles of sorghum flours, at the same time underlying the importance of particle size and variety. This information could be helpful for processors to better decide the destination of sorghum flours to obtain further nutritious foods.

Author Contributions: All authors contributed equally to this research. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by "Stefan cel Mare" University of Suceava.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable. **Data Availability Statement:** Not applicable.

Acknowledgments: This research was supported by "Stefan cel Mare" University of Suceava.

Conflicts of Interest: The authors declare no conflict of interest.

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Appl. Sci. **2021**, 11, 11881 17 of 17

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