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Physical and chemical characterization of *Corchorus olitorius* leaves dried by different drying techniques

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Abstract

Molokhia, *Corchorus olitorius*, is a popular leafy vegetable, known in many world regions as a good source of nutritional and medicinal properties. Due to its short shelf life and the limited harvesting time, processing such as drying techniques permit to preserve and provide it throughout the year. In the present study, it was attempted to reveal the main physical and chemical characteristics of molokhia leaves. Also, three drying techniques, shade drying (SHD), convective drying (COD), and microwave drying (MID), have been applied to study the kinetics and their main physical and chemical effects. The analysis demonstrated that molokhia leaves are a good source of phenolic compounds, flavonoids, and chlorophylls pigments. Those bioactive compounds have provided the leaves with considerable antiradical scavenging and reducing capacities. Drying time decreased from days, in the case of SHD, to some hours when using COD, and less than 20 min when using MID. Increasing drying temperature and power input have increased the drying rate. Modelling of drying kinetics of MID three power inputs (350, 500 and 750 W) and COD at 60 °C exhibited a high fitting for most empirical models ($R^2 > 0.980$). SHD was less deleterious on leaves colour. Also, it preserved the content of phenolics, flavonoids, and thus the antioxidant activity of leaves. On the contrary, COD at 80 °C had a detrimental effect on previous components and their activity. Vega-Gálvez model can be presented as the best-fitted model to describe the rehydration kinetics of dried leaves. Rheological analysis of the aqueous extracts of the leaves demonstrated the effect of time and grinding on the increase of mucilage diffusion. The obtained results could help industrials to choose the convenient drying method and more analysis on the subject are recommended.

Keywords Molokhia leaves · Antioxidants · Drying techniques · Modelling · Viscosity

1 Introduction

Corchorus olitorius, molokhia, jute mallow or simply jute, is a leafy vegetable that belongs to Malvaceae family. It is native to tropical Africa, Asia, and now is spread out over the world. Their leaves are consumed and largely used in folklore medicine, believing in their high nutritional and medicinal properties against as a series of ailments and diseases. It is used fresh or as dried leaves in soups and broths in many Arab and African regions [1–3]. About 95% of the world molokhia plant grow in South Asia, specifically in India and Bangladesh. It is used for the production of fibres in textiles and their by-products are included in paints, cosmetics, medicine, and other uses [4]. Molokhia is a widely used plant with an outstanding value due to its agronomic benefits and contribution to local economies [5].

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Various studies have evaluated the biochemical compositions and the main biological activities of molokhia. Molokhia leaves have been reported to be a good source of bioactive compounds such as vitamin C, α-tocopherol, and phenols such as chlorogenic acid, quercetin glycosides, caffeic acid, isorhamnetin [6, 7]. Dry oils of leaves and stems were analysed and revealed that they contain a high percentage of 2, 4-di-*tert*-butylphenol and fatty acids such as hexadecenoic and ethyl palmitate acids [8]. These bioactive compounds enter into many biological activities and also processes of attenuation of free radicals which are among the causes of degenerative diseases. Therefore, a large list of the positive activities is linked to their various bioactive compounds, such as the interesting antioxidant capacity, enzyme inhibitory activity, and antimicrobial activity [6–9]. In addition to its medicinal properties, molokhia contains a considerable amount of minerals and proteins, which make it a nutritive plant [6, 8].

Molokhia leaves, and as mentioned above, can be eaten fresh or transformed (drying, extraction…) into many products. Also, it can be incorporated into a list of products to enrich them and improve not only the organoleptic quality, but also their nutritional profile and functional properties [10, 11]. Post-harvest processing can influence the quality, composition, and content of bioactive compounds, and subsequently the nutritive value and biological activities (antioxidant, antibacterial). Therefore, various studies have analysed the effect of different variables on the physicochemical stability of molokhia leaves [9, 11–14].

Drying is a preservation process widely used for various food products (meats, dairy products, fruits, and vegetables). Leafy vegetables are similarly perishable products, the application of dehydration processes increases their shelf lives by the reduction of moisture contents, and consequently the reduction of undesirable chemical reactions and microbial activities. Furthermore, drying can also reduce the high costs of transportation and storage. It can be performed by different traditional techniques, such as sun drying, shade drying and oven drying. Various sophisticated techniques such as microwave drying, infrared drying, and freeze-drying have attracted more attention for the application due to their new advantages. While microwave drying and infrared drying require a short processing time, which means low energy costs, they are still far from the high product quality of the freeze-drying process, which endures long hours [14–18].

Dried herbs can be widely applied in several sectors (food, pharmaceutical, cosmetics, and other industries) as flavouring, medicinal, and dyeing agents [10, 19, 20]. Mostly, temperature, time, and the applied energy powers were among the affecting factors which influence the chemical compositions (nutrients, phenolic compounds, vitamins, etc.) and the final beneficial activities (e.g., antioxidant activity) of processed products [9, 13, 19–21]. Therefore, studying the effect of different drying techniques and evaluating the engendered effects could help to choose the suitable technology and of course their main parameters, to preserve the composition and the beneficial functionality of plant materials. Moreover, colour, flavour and texture should be taken into consideration to obtain high quality products.

The objective of this study is to compare the different drying techniques and the parameters on the quality of molokhia leaves. Numerous analyses, such as total phenolic, flavonoid, and antioxidant activities, were performed to assess the impact of drying. In addition, the drying kinetics, the rehydration capacity, and the rheology of the extracts of the dried leaves were studied.

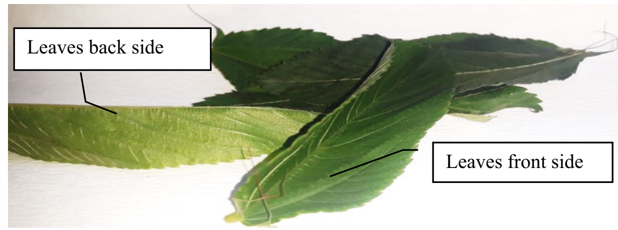
2 Materials and methods

2.1 Material and chemical

Molokhia leaves (Fig. 1) were purchased from the local market in Samsun, Turkey. Leaves were sorted and colour-darkened and damaged ones were discarded. They were immediately kept at 4 °C for till further treatments and analyses.

2,4,6-Tris(2-pyridyl)-1,3,5-triazine (TPTZ), 2,2-Diphenyl-1-Picrylhydrazyl (DPPH). 6-hydroxy 2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox), 2,6-dichlorophenolindophenol, hydrochloric acid (HCl, 37%), methanol (99,8%), acetone, sodium nitrite, sodium hydroxide (NaOH), (–)-epicatechin (Ep), were purchased from Sigma-Aldrich. gallic acid (GA) and sodium carbonate from Riedel-de Haen. Potassium chloride, sodium acetate, glacial acetic acid, from CARLO ERBA. Aluminium chloride, Folin–Ciocalteau, iron chloride (Merck), BHT (Butylated hydroxytoluene) (SAFC), analytical balance (Radwag, AS 220/C/2, Poland), precision balance (Radwag, PS 3500.R1, Poland), drying oven (NÜVE, FN 500P, Turkey), shaking water bath (Model ST 30, NÜVE. Turkey) were used in this study.

Fig. 1 Molokhia leaves (*Corchorus olitorius*)



2.2 Physical and chemical analyses

Water activity (a_w), for fresh and dried molokhia leaves was determined using a calibrated water activity meter at 25 °C ± 0.1 (Aqualab, 4TE, USA). To determine moisture and dry matter, a homogenized quantity of leaves (ca. 3 g) was taken and dried at 70 °C till a constant weight. The CIE L*a*b* scale was used to measure leaves colour (front = adaxial and back = abaxial) using a digital colorimeter (Model CR-400, Minolta-Konica Sensing Inc., Osaka, Japan) and to calculate colour change ΔE (Eq. 1). The length and width of leaves are measured by digital calliper (TRESNA, Series: EC16, China). The pH of fresh leaves was measured using pH-meter (Oakton pH 700 Benchtop Meter). A previously described method was used for the determination of titratable acidity (Sadler and Murphy 2010). The concentration of acid is expressed as % of citric acid.

$$\Delta E = \sqrt{(L_i^* - L_0^*)^2 + (a_i^* - a_0^*)^2 + (b_i^* - b_0^*)^2}, \quad (1)$$

where, L_i, a_i, b_i, are lightness, greenness, and yellowness of treated leaves, and L_0, a_0, b_0, are lightness, greenness, and yellowness of fresh leaves, respectively.

2.2.1 Extracts preparation

To prepare the methanolic extract, 0.5 g and 1 g of dried and fresh leaves, respectively, were extracted in 20 ml of 70% methanol. The extraction lasted for 20 min in ultrasonic bath (Wisd. Wise Clean, Kore) at 20 °C ± 2.

To prepare water extract for rheological measurements, 0.5 g of 60 °C dried leaves (whole and grinded leaves) were extracted using distilled water (20 ml) at 50 °C ± 1. To understand the effect of extraction time on the two leaves, the time was divided to 0.5, 1, 2, 3, 4, 5, 6, 12 and 16 h. After extraction, and to remove insoluble and suspended particles the samples were centrifuged at 5000 rpm, and 20 °C for 15 min using 380R Hettich centrifuge (Germany).

Chlorophyll a and b contents were spectrophotometrically determined using the previous procedure of Jeffrey and Humphrey [22] with some modification. Using the methanolic extracts, the amount of total phenolic compounds (TPC) was measured spectrophotometrically at 760 nm according to the Folin-Ciocalteu method [23]. Flavonoid content (TFC) was determined using a spectrophotometric method based on the aluminium complex formation [24]. The free radical scavenging capacity was assessed by using Diphenyl-1-Picrylhydrazyl (DPPH) as a free radical [25]. The reducing capacity (FRAP) of berries extracts was measured according to the method of Benzie and Strain [26]. Minor modifications are done for many analyses. Calibration curves are used to calculate concentrations, curves prepared by the corresponding standards (Trolox equivalent, TE; GAE, Gallic acid equivalent; Epicatechin equivalent, EpE) showed high values of determination coefficient ($R^2 > 0.990$).

2.3 Drying techniques and modelling

Molokhia leaves were dried using three methods (Fig. 2). Shade-drying (SHD): fresh leaves were air-dried in shade at laboratory temperature (≈ 25 °C) for 48 h. Convective drying (COD): leaves were dried in a laboratory-scale convective dryer (Eksis Makina. Turkey) for 3 different temperatures (40, 60 and 80 °C) at 0.75 m/s (airspeed) and 3 rpm (trays rotation). Microwave drying (MID): leaves were dried till constant weight, using microwave (Arçelik MD 581 Model, Turkey) and at different powers (350, 500 and 750 W). All drying conditions were done in triplicate (sample weight was 50 or 100 g).

Drying curves for 60 °C and all microwave dried samples were modelled according to the empirical models in Table 1. Moisture ratio (MR) versus time (h/min) of experimental data was fitted with previous models. MR is dimensionless and calculated as following:

Fig. 2 Drying techniques

Table 1 Empirical models applied to drying curves

	Model name	Model equation	References
1	Newton	MR = exp(− kt)	[27]
2	Henderson and Pabis	MR = a exp(− kt)	[28]
3	Logarithmic	MR = a exp(− kt) + b	[27]
4	Two-term	MR = a exp(− kt) + b exp(− gt)	[27]
5	Noomhorm and Verma	MR = a exp(− kt) + b exp(− gt) + c	[29]
6	Modified Henderson and Pabis	MR = a exp(− kt) + b exp(− gt) + c exp(− ht)	[30]
7	Two-term exponential	MR = a exp(− kt) + (1 − a) exp(− kat)	[27]
8	Diffusion Approximation	MR = a exp(− kt) + (1 − a) exp(− kbt)	[30]
9	Verma et al	MR = a exp(− kt) + (1 − a) exp(− gt)	[31]
10	Page	MR = exp(− kt^n)	[32]
11	Wang and Singh	MR = 1 + (at) + bt^2	[33]
12	Logistic	MR = b/(1 + a exp(kt))	[34]
13	Midilli	MR = a exp(− k(t^n)) + (bt)	[32]

MR moisture ratio(dimensionless), *t* time, a, b, c and k = model constants

$$MR - \frac{M(t) - M_e}{M_0 - M_e}, \tag{2}$$

where, M(t) is the moisture content in dry basis at time (h/min), M_e is the moisture content in dry basis at equilibrium and M_0 is the initial moisture content (t = 0).

3 Rehydration

Rehydration was done only for 60 °C dried samples to evaluate the behaviour of leaves. Dried leaves were immersed in distilled water of 50 °C at a ratio of 1:40. The weighing was done in duplicate at 0, 30, 60, 120, 180, 240, 360, 720, and 960 min. Rehydrated leaves were gently blotted with tissue paper before weighing to remove the excess of surface water.

$$M(t) = \frac{W(t) - W_0}{W_0}, \tag{3}$$

where, M(t) is the moisture content at t time kg/kg dry basis (d.b), W(t) is the sample weight (kg) at t time, W_0 is the sample weight (kg) at t = 0 min,

Rehydration fitting curve (60 °C dried leaves) was modelled according to the empirical models in Table 2.

The modelling was coded using software MATLAB (2016a). The coefficient of determination (R^2), root mean square error (RMSE), sum of square error (SSE) and Chi-square (X^2) were all calculated to show the adequacy of models and the goodness of fitting between experimental data and predicted values. The best models are evaluated according to the highest value of R^2 and the lowest values of RMSE, SSE, and X^2.

Table 2 Empirical models applied to rehydration curve

	Model name	Model equation	References
1	Peleg	$M(t) = M_0 + (t/(a + bt)))$	[35]
2	Exponential model	$M(t) = (M_0 + ((M_e - M_0)\ exp(-\ a\ t\text{^}k)))$	[36]
3	Exponential related equation	$M(t) = M_e\ (1 - exp(-\ a\ t))$	[37]
4	Weibull	$M(t) = M_e + (M_0 - M_e)\ exp(-\ ((t/b)\text{^}a))$	[38]
5	First-order kinetic	$M(t) = M_e + (M_0 - M_e)\ exp(-\ a\ t)$	[39]
6	Vega-Gálvez model	$M(t) = a\ exp(-\ b/((1 + t)\text{^}k))$	[38]

M(t) = the moisture content at t time kg/kg d.b; M_0 = the moisture content (kg/kg d.b) at t = 0; M_e = the moisture content (kg/kg d.b) at equilibrium (estimated after calculation of Peleg model, where $M_e = 1/b$); t = time; a, b and k = model constants

3.1 Rheological properties

Rheological measurements were carried out using Haake Mars Rheometer (Thermofisher Scientific) and P35 TiI parallel-plate as a measuring geometry with a gap of 1 mm. The rheological properties of water extracts (whole and grinded leaves) at different hours, were studied at an increasing shear rate (0.1–100 S^{-1}) in 180 s under 25 °C. Apparent viscosity and flow curves were obtained. Apparent viscosity (mPa.s) was calculated as the mean of all viscosities between a shear rate of 51 and 100 s^{-1}, which were almost constant. The flow curves were fitted to Power-law model (Eq. 4) and the goodness of modelling fitting was evaluated by R^2 and RMSE. All measurements were done in duplicates.

$$\sigma = K\gamma^n, \tag{4}$$

where σ is the shear stress (mPa), K is the consistency index (mPa.s^n), γ is the shear rate (s^{-1}) and n is the flow behaviour index (dimensionless).

3.2 Statistical analysis

Statistical significance ($p < 0.05$) between different techniques of drying and groups of time was analysed through analysis of variance (ANOVA) using SPSS statistics software (version 23). The comparison of means was evaluated by Tukey test.

4 Results and discussion

4.1 Characteristics of fresh molokhia leaves

The physical and chemical properties of molokhia leaves are shown in Table 3. The analysis demonstrated that lanceolate shaped and green molokhia leaves are considerably large leaves and have a high water activity and content of moisture. Dry matter, water activity, length and width were 25.47%, 0.989, 78 mm and 31 mm, respectively. The dry matter found

Table 3 Physical and chemical characteristics of molokhia fresh leaves

		Mean	± S.D		Mean	± S. D
Dry matter(%)		25.47	0.67	Water activity, a_w	0.989	0.0074
Length (mm)		78.00	11.22	pH	7.08	0.08
Width (mm)		31.03	3.64	TA% (citric acid)	0.58	0.05
Leaf front colour	L *	38.55	2.25	Chlorophyll *a* (mg/100g FW)	60.33	5.64
	a*	− 14.78	1.61	Chlorophyll *b* (mg/100g FW)	18.39	2.17
	b*	19.06	2.55	FRAP (mMol TE/g FW)	164.00	17.28
Leaf back colour	L *	50.89	1.56	DPPH (mMol TE/g FW)	15.94	5.76
	a*	− 13.76	0.61	TPC (mg GAE/g FW)	45.73	13.14
	b*	18.66	0.87	TFC (mg EpE/g FW)	2.87	0.59

was close to that of Yakoub et al. [9], Chen and Saad [14], and Mutuli and Mbuge [13], who found 22.47%, 25.4%, and 26.20%, respectively. Moreover, the colour parameters L*a*b* for both leaf sides displayed similar high green pigmentation (front a* = − 14.78, back a* = − 13.76) and an important yellowness (front b* = 19.06, back b* = 18,66). However, the leaf back side presented more brightness than the front side (front L = 38.55, back L = 50.89). These findings are very close to that of Alimi et al. [2] who found 36.93, − 10.34, 18.00, for L*, a* and b*, respectively. The high greenness of leaves is attributed to the high content of total chlorophylls, in which the chlorophyll a and b were 60 mg/100 g and 18 mg/100 g of fresh weight, respectively. Various chlorophyll contents of molokhia leaves were found in literature, which could be related to the difference in the varieties themselves, or due to environmental conditions and agricultural practices, or the methods of extraction and determination [3, 11]. The pH of leaves was neutral (7.08) and the acidity displayed very low value (0.58%, Citric acid). The pH values are generally proportional to the content of acids [15].

Fresh leaves are an important source of phenolic compounds, they contain up to 46 mg GAE/g FW of total phenolics and 2.87 mg EpE/g FW of total flavonoids. Due to these and other compounds, methanolic extracts have shown an important reducing capacity and radical scavenging power, where FRAP and DPPH tests showed 164 mMol TE and 16 mMol TE/g FW, respectively. Many recent studies have reported that molokhia contains a high content of phenolic compounds and an interesting antioxidant capacity. Oboh et al. [7] found that total phenolic compounds, free and bound phenolics, were 389 mg/100 g and 450 mg/100 g, respectively. Chlorogenic acid, caffeic acid, and isorhamnetin were the dominant phenolic compounds. Also, they found that the antiradical capacity exceed 14.5 mmol TE/g. Oboh et al. [1] analysed the hydrophilic extracts of molokhia and have found 631 mg/100 g and 228 mg/100 g dry weight for total phenolic and flavonoid, respectively. The variation of phenolic profile from molokhia leaves may be influenced by the season, minerals, micronutrients or by the extraction solvents [3, 9]. Beside the chemical composition of molokhia leaves and their importance for human health, the physical characteristics such as colour and freshness are important factors for consumers to select this vegetable.

4.2 Drying kinetics

In order to preserve the quality and prolong the shelf life, various drying techniques have been used to reduce the water activity and the moisture content of molokhia leaves. The ways of action variate from a technique to another, also dependent tightly to the applied parameters (temperature, time, power input, etc.). In the present study, MID was the fastest way of drying less than 20 min for less than 100 g. However, COD was slower compared to the previous technique. The low temperature (40 °C) was the slowest and took some hours to reach low moisture content. In contrary, the increase of temperature to 60 °C and also to 80 °C make it faster. SHD was the slowest technique in this study. This observation makes sense since no energy (radiation, convection) or air velocity was applied, just the room environment. SHD technique could last days because of the natural conditions, weak temperature and absence of air velocity [40, 41].

The effect of microwaves on the drying kinetics of molokhia leaves was analysed at three power inputs (350, 500 and 750 W). Figure 3, obtained from plotting moisture content against drying time, clearly shows the variation of drying rate between the three powers. The high microwave power, 750 W, reached the plateau several minutes before 500 W and much more before 350 W. It is very clear that the lower the drying energy, the slower the drying rate. Microwaves apply electromagnetic waves to water molecules in wet materials. Due to their bipolarity, water molecules begin to rotate rapidly, resulting in the production of heat through friction movements. The increase in the of power input speeds up the friction movements and consequently the production of heat. Thus, and in turn, the produced heat accelerates the diffusion rate of water and its evaporation [42, 43].

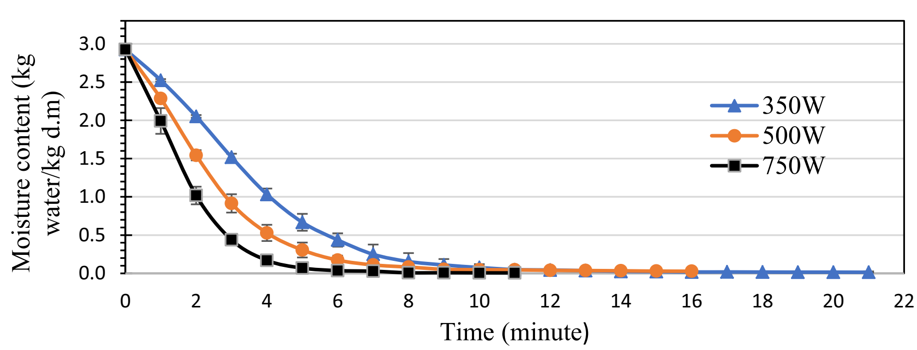


Fig. 3 Kinetics of moisture content of molokhia leaves, microwave-dried at 350 W, 500 W and 750 W

The results of the modelling, using various empirical models, of the curves obtained previously for MID inputs as well as for convection drying at 60 °C are described in the Tables 4 and 5. The three drying inputs had many best-fitted models with a high R^2 (> 0.990) and low values of RMSE, SSE, and X^2. They presented in common models of Modified Henderson and Pabis, Two-term exponential, and Page model. Meanwhile, COD at 60 °C had high fitting with all models ($R^2 > 0.980$) except the Midilli model ($R^2 = 0.544$).

The high values of the correlation coefficients imply a good correlation between the empirical models and the experimental drying kinetics. Therefore, most of these models are reliable and suitable for predicting the moisture content of molokhia leaves using MID and even under COD. Several work on modelling of drying of molokhia and other leaves, using different drying techniques, presented similar results and the reliability of the different models to predict drying [30, 44, 45].

4.3 Colour evaluation after drying treatments

Molokhia leaves colour has been studied to assess the variation of colour after drying process using SHD, COD (40 °C, 60 °C, 80 °C) and MID (350 W, 500 W, 750 W). The obtained results are shown in Fig. 4.

The lightness (L*) of fresh and dried leaves are shown in Fig. 4a. Leaves front side did not show any significant difference ($p < 0.05$) between before and after different drying methods. However, leaves back side demonstrated that shade and 40 °C dried kept their fresh brightness, meanwhile, the microwave dried ones were more affected and showed the lowest values.

For both leaf sides, there was a significant decrease of greenness (a*) for all dried samples (Fig. 4b). Leaves front side had the most pronounced decrease especially at three COD temperatures. For leaf back side, COD temperatures at 80 °C had affected more than other temperatures. MID at 750 W was the least affecting drying method for both sides.

In general, as shown in Fig. 4c, the yellowness (b*) of leaves back side have been significantly ($p < 0.05$) increased comparing to the fresh leaves. However, the front side showed a decreasing of yellowness at 60 °C and 80 °C temperatures. MID at 750 W gave the highest yellowness for both sides.

ΔE calculation describes the colour change amplitude in a general way. As shown in Fig. 4d, ΔE have demonstrated no statistically significant ($p > 0.05$) effect for all drying methods on the colour of leaves front side. However, MID at 350 W and 500 W had highest effect on the colour of leaves back side. This could be probably attributed to the effect of MID power on leaves colouring agents. Despite the long treatment time, COD at 40 °C and 60 °C followed by SHD had the lowest drying effect on leaves colour.

Several studies have demonstrated the effect of processing on the colour of molokhia leaves. They found that the greenness, main colour of leafy vegetables, was affected by cooking, blanching and by different methods of drying. The colour change was highly correlated with the chlorophyll content. Drying temperature and duration were the crucial factors in pigments retention, the high heat treatment causes the oxidation and the degradation of chlorophylls [2, 11, 15, 44]. Thus, the application low heat and short time treatment could retain the pigments and consequently the fresh-like colour and the quality of dried leaves. In the present work, SHD at 40 °C and MID at 750 W were good examples for low temperature and short time treatments, respectively.

4.4 Drying effect on phenolic compounds, flavonoids, and antioxidant activity

As shown in Table 6, drying techniques and different conditions have shown a great variation in antioxidants content and their capacity and also some significant difference between dry matters. The observed difference in dry matter could be related to a very low residual moisture. SHD showed the high content of total phenolics and flavonoids, which reached, 227 GAE/g and 13.5 EpE/g, respectively. In contrast, COD at 80 °C gave the weakest values many times lower than SHD. The increase of drying temperature from 40 °C to 60 °C, and finally to 80 °C, proves the negative effect of heat on phenolics and flavonoids. Also, MID for all three powers, had an effect on the phenolic content. Similar to 40 and 60 °C, microwave dried leaves demonstrated a significant decrease ($p < 0.05$) of flavonoids, but not pronounced comparing to 80 °C. Despite the high applied energy power, MID at 750 W had a negligible effect on flavonoids. It seems that MID energy does not affect the flavonoids as much as the duration of treatment.

Many works found variation in final dry weights that was related to residual moisture, despite this they found that the effect of drying methods and conditions were the main factors affecting final leaf characteristics (color, bioactive compounds, antioxidant capacity, extraction, rehydration…) [42–49]. Low-temperature drying processes such as freeze, room, and shade drying are good examples of processes that result in high residual moisture and retention of bioactive

Table 4 Results of the statistical analysis of microwave drying data at 350 W and 500 W

Models	350 W					500 W				
	R^2	SSE	RMSE	X^2	Variables	R^2	SSE	RMSE	X^2	Variables
Newton	0.974	0.051	0.049	0.00242	k = 0.274	0.985	0.023	0.038	0.00144	k = 0.391
Henderson and Pabis	0.981	0.038	0.044	0.00191	a = 1.089, k = 0.295	0.987	0.019	0.035	0.00125	a = 1.057, k = 0.410
Logarithmic	0.984	0.032	0.041	0.00168	a = 1.106, b = − 0.026, k = 0.274	0.989	0.016	0.034	0.00116	a = 1.070, b = − 0.018, k = 0.389
Two-term	0.987	0.025	0.037	0.00138	a = -5.590, b = 6.665, g = 0.193, k = 0.179	0.987	0.019	0.038	0.00143	a = − 16.8, b = 17.854, g = 0.389, k = 0.388
Noomhorm and Verma	0.984	0.032	0.043	0.00188	a = -17.402, b = 18.501, c = − 0.025, g = 0.263, k = 0.262	0.990	0.014	0.035	0.00119	a = − 11.455, b = 12.521, c = − 0.008, g = 0.317, k = 0.310
Modified Henderson and Pabis	0.994	0.011	0.026	0.00069	a = 115.075, b = − 114.257, c = 0.244, g = 0.508, h = 0.293, k = 0.506	0.999	0.000	0.005	0.00003	a = 1.739, b = − 9.639, c = 8.90, g = 0.985, h = 0.959, k = 0.578
Two-term exponential	0.998	0.003	0.012	0.00016	a = 2.027, k = 0.424	0.999	0.000	0.005	0.00002	a = 1.965, k = 0.589
Diffusion Approximation	0.983	0.033	0.042	0.00175	a = 6.286, b = 0.920, k = 0.174	0.989	0.016	0.034	0.00113	a = − 67.719, b = 1.005, k = 0.271
Verma et al	0.999	0.002	0.010	0.00010	a = 11.254, g = 0.582, k = 0.531	0.989	0.017	0.034	0.00118	a = − 12.466, g = 0.286, k = 0.279
Page	0.999	0.001	0.006	0.00003	k = 0.129, n = 1.508	0.999	0.000	0.005	0.00002	k = 0.251, n = 1.392
Wang and Singh	0.924	0.150	0.086	0.00748	a = -0.153, b = 0.005	0.895	0.155	0.102	0.01037	a = − 0.204, b = 0.009
Logistic	0.981	0.038	0.045	0.00201	a = 5496.713, b = 5988.759, k = 0.294	0.999	0.000	0.005	0.00003	a = 0.407, b = 1.412, k = 0.715
Midilli	0.999	0.001	0.006	0.00003	a = 0.993, b = 0.0001, k = 0.125, n = 1.529	0.999	0.000	0.004	0.00002	a = 1.001, b = 0.0003, k = 0.251, n = 1.398

Table 5 Results of the statistical analysis of microwave drying data at 750 W and convective drying at 60 °C

Models	750 W					60 °C				
	R^2	SSE	RMSE	X^2	Variables	R^2	SSE	RMSE	X^2	Variables
Newton	0.984	0.019	0.041	0.00169	k = 0.5523	0.985	0.061	0.036	0.00127	k = 0.046
Henderson and Pabis	0.986	0.017	0.041	0.00167	a = 1.040, k = 0.570	0.990	0.039	0.029	0.00084	a = 1.081, k = 0.049
Logarithmic	0.988	0.014	0.039	0.00155	a = 1.059, b = − 0.023, k = 0.535	0.993	0.027	0.024	0.00060	a = 1.094, b = − 0.031, k = 0.045
Two-term	1.000	0.000	0.004	0.00001	a = − 10.284, b = 11.283, g = 1.060, k = 1.159	0.993	0.030	0.026	0.00066	a = 1.119, b = − 0.119, g = 10.261, k = 0.051
Noomhorm and Verma	0.934	0.078	0.106	0.01119	a = 0.843, b = 37.484, c = − 37.451, g = 0.0003, k = 0.370	0.997	0.010	0.015	0.00023	a = 12.165, b = − 11.148, c = − 0.005, g = 0.079, k = 0.075
Modified Henderson and Pabis	0.991	0.010	0.042	0.00172	a = 0.288, b = 69.037, c = − 68.318, g = 0.815, h = 0.820, k = 0.550	0.998	0.010	0.015	0.00022	a = 0.028, b = − 7.011, c = 7.987, g = 0.086, h = 0.078, k = 0.539
Two-term exponential	0.999	0.000	0.005	0.00003	a = 2.036, k = 0.855	0.985	0.061	0.036	0.00130	a = 0.0001, k = 388.216
Diffusion Approximation	0.990	0.012	0.036	0.00130	a = 8.124, b = 0.943, k = 0.358	0.985	0.060	0.036	0.00130	a = 1.352, b = 0.914, k = 0.045
Verma et al	0.989	0.012	0.037	0.00138	a = − 9.951, g = 0.386, k = 0.372	0.998	0.010	0.015	0.00021	= − 16.500, g = 0.078, k = 0.081
Page	0.999	0.000	0.003	0.00001	k = 0.387, n = 1.446	0.998	0.010	0.014	0.00021	k = 0.020, n = 1.258
Wang and Singh	0.916	0.099	0.099	0.00988	a = − 0.294, b = 0.019	0.972	0.115	0.049	0.00244	a = − 0.029, b = 0.0002
Logistic	0.999	0.000	0.003	0.00001	a = 0.342, b = 1.343, k = 1.057	0.998	0.010	0.014	0.00021	a = 0.726, b = 1.729, k = 0.071
Midilli	0.880	0.141	0.133	0.01760	a = 0.999, b = 0.003, k = 0.390, n = 10.721	0.544	1.861	0.203	0.04136	a = 0.620, b = − 0.008, k = 24.936, n = − 54.889

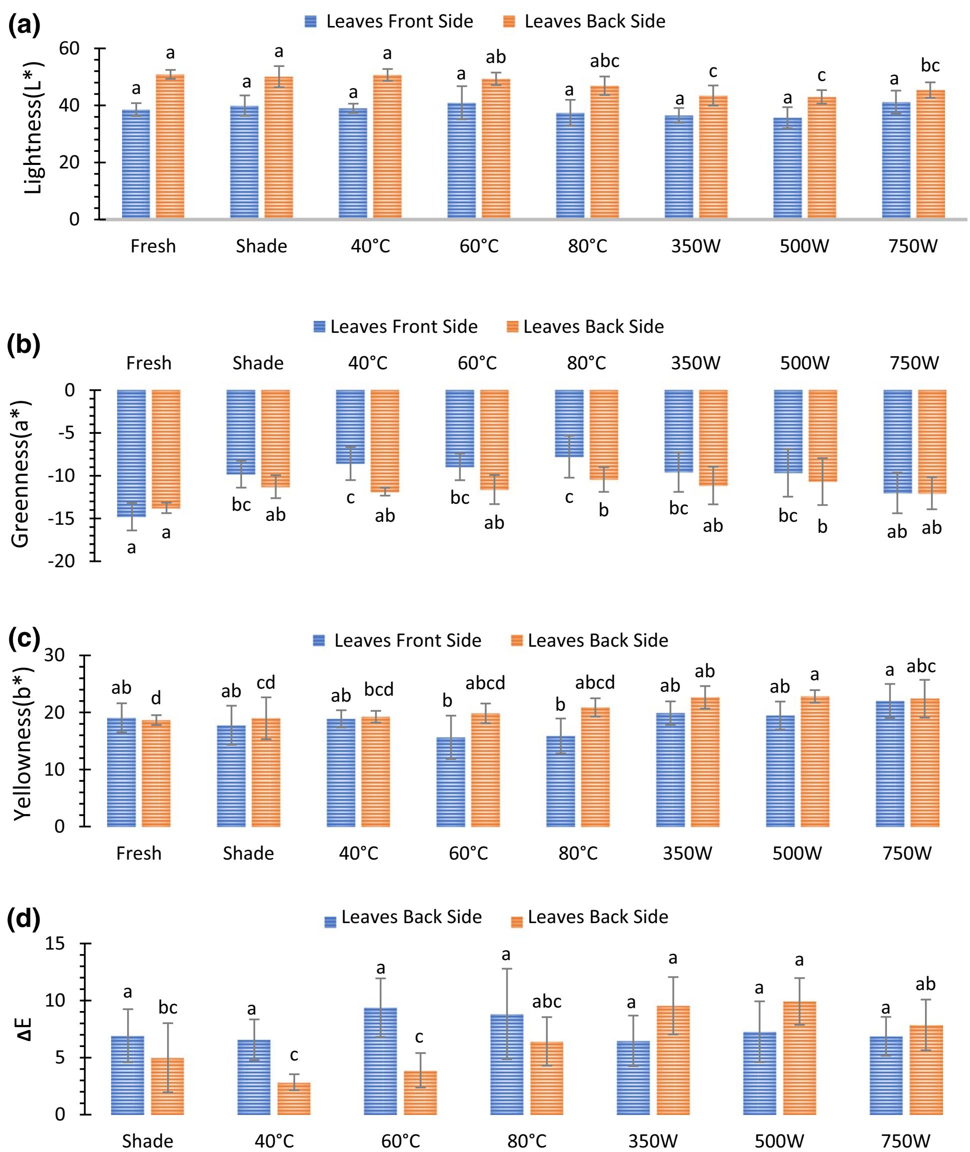


Fig. 4 Drying techniques and parameters effect on colour coordinates (**a**: Lightness, **b**: greenness, **c**: yellowness, **d**: ΔE) of front and back sides of molokhia leaves. Bars with different letters are significantly different ($p < 0.05$)

Table 6 Drying techniques and their parameters variation effect on dry matter, water activity, antioxidant compounds contents and their related antioxidant activities

Drying technique	Total phenolic compounds (mg GAE /g d.b)	Total flavonoids (mg EpE/g d.b)	DPPH (mMol TE/g d.b)	FRAP (mMol TE/g d.b)	Water activity	Dry matter %
Shade	227.1 ± 34.1^{a}	13.5 ± 1.7^{a}	157 ± 7.9^{a}	442.6 ± 10.6^{a}	0.499 ± 0.025^{d}	28.27 ± 0.27^{a}
40 °C	210.4 ± 25.9^{ab}	9.7 ± 0.4^{c}	$157.9 \pm 22.^{1a}$	397.4 ± 37.3^{a}	0.558 ± 0.027^{a}	23.95 ± 0.24^{b}
60 °C	164.4 ± 36.1^{ab}	10.1 ± 1.4^{bc}	143.6 ± 8.4^{a}	374.7 ± 7^{ab}	0.529 ± 0.011^{abc}	26.04 ± 0.25^{ab}
80 °C	79.4 ± 10.8^{c}	2.2 ± 0.1^{d}	27.2 ± 9.1^{b}	212.7 ± 62.6^{d}	0.527 ± 0.011^{bcd}	27.95 ± 2.74^{a}
350 W	153.9 ± 1.5^{ab}	8.6 ± 0.3^{c}	121.6 ± 15.3^{a}	266.7 ± 15.9^{cd}	0.509 ± 0.005^{cd}	25.85 ± 0.26^{ab}
500 W	175.3 ± 48.6^{ab}	9.9 ± 1.2^{bc}	148.2 ± 6.2^{a}	304.7 ± 43.3^{bc}	0.541 ± 0.004^{ab}	$26.19 \pm 0.14a^{b}$
750 W	143.1 ± 39.8^{bc}	12.8 ± 2.2^{ab}	142.5 ± 37.4^{a}	275.8 ± 18.8^{cd}	0.544 ± 0.007^{ab}	25.49 ± 0.59^{ab}

Values followed by the same letter (s) are not significantly different at $p \le 0.05$ according to the Tukey's test compounds. Additionally, exposure to treatment processes was controlled by stabilization of weights, meaning that this stabilization is an outcome related to the type and drying conditions applied.

Also, the evaluation of antioxidant activity using DPPH and FRAP tests gave the same trend as total phenolics and flavonoids. COD at 80 °C recorded the lowest values for both DPPH and FRAP analysis. In general, the rest of the drying techniques seemed to have no significant difference for DPPH, however, in the FRAP analysis, SHD had the highest values, followed by COD (40 °C and 60 °C), and finally by MID. This indicates clearly the high correlation between the antioxidant potency and the content of phenolic compounds. Additionally, total phenolic compounds and flavonoids are sensitive compounds influenced by heat, energy output, and drying time, which in turn affect their antioxidant and scavenging activities.

All the dried leaves have shown a high content of total phenol compounds comparing to the fresh leaves. This finding are in agreement with previous work of [12], who found that sun drying have increased the total phenol content compared to the fresh leaves of leafy vegetables including molokhia leaves. The increase of total phenol content may be attributed to the breakdown of some compounds such as tannins and the liberation of more phenols. Despite of the decrease of vitamin C content, the increase of phenols has increased the reducing capacity and free radicals scavenging ability, which proves that phenols are potent antioxidant phytochemicals [12].

Many studies have shown the negative effect of increasing temperature on the antioxidative compounds and the final antioxidant capacity, represented by reduction potential and scavenging radicals (DPPH, FRAP, ABTS…). It has been found that the thermal processing, cooking, blanching and drying, have affected the final antioxidant potential of molokhia broths [2]. Mutuli and Mbuge [13] found that the effect of drying temperature on molokhia and cowpea leaves was more profound than drying time. The increase of temperature from ambient to highest degrees (100 °C) has increased the deterioration of least stable nutrients (vitamin C). Shitanda and Wanjala [15] studied the effect of drying methods on molokhia leaves and using vitamin C as a quality factor. Similar to our finding SHD is considered as the best drying method after freeze-drying, compared to sun and vacuum drying. SHD is mostly chosen because of its low costs.

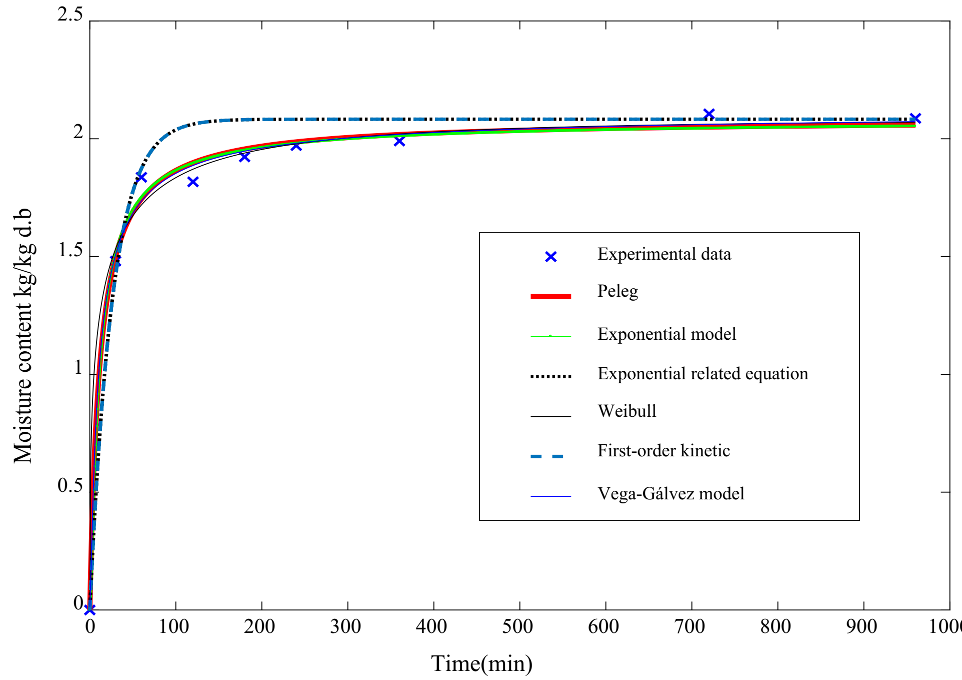
Many works have found an opposite trend, where increasing the temperature increased the retention of the total content of total phenolics and flavonoids. This result is explained by the effect of the long-lasting treatment, in which drying at low temperatures takes much longer to reach the lowest moisture levels [11]. Another study of Hamrouni-Sellami et al. [21] presented another opposite result, where the 800 W microwave dried sage plants gave the highest values of phenolic content, many times greater than the fresh plant. They justified this increase by the disruption of plant tissue and the liberation phenolic compounds. However, the drying of this plant at 45 °C using infrared had low values less than ambient air shade drying (22 °C) and 600 W. They found also a similar result to our present study, that the total flavonoids content has increased when using ambient air shade and MIDs. The increase in temperature to 65° C had the lowest values of flavonoid contents. This can confirm the finding of the present work, in which increasing the drying temperature above 60° C (80° C) causes a profound effect on the composition of flavonoids.

Hamrouni-Sellami et al. [21] studied DPPH radical scavenging, β-carotene bleaching, and ferric-reducing antioxidant potential (FRAP) of dried sage plants. The highest radical scavenging activity was encountered with MID at a power of 800 W, whereas oven dried plant at 65 °C recorded the lowest activity. Generally, they found that the scavenging activity increased as microwave output power and infrared temperature increased, however there was a decrease when oven drying temperature increased. In addition, Orphanides et al. [19] found that freeze-drying of spearmint leaves had the highest retention of phenolic compounds, followed by sun drying, oven drying, and finally MID. Evaluation of hydroxycinnamic acid showed the same trend with total phenolic compounds. Additionally, the antioxidant capacity assessment (DPPH) revealed a similar trend and ensured that freeze-drying preserved the quality of spearmint leaves in terms of antioxidant potency.

4.5 Rehydration fitting

The rehydration behaviour was analysed for molokhia leaves dried at 60 °C. The rehydration kinetics are plotted and represented in the Fig. 5. The water absorption process of the dried leaves was very rapid within the first hour, with almost 1.5 kg of moisture/kg of dry matter. Then the rehydration rate begins to decrease for up to 5 h of the process. A few hours later, the rehydration process reaches the plateau at a level of 2 kg of moisture /kg of dry matter. Beyond rehydration environmental conditions (water content, rehydration temperature), the rate of rehydration during the process and the final capacity are strongly affected by the cell structure changes that have occurred in the leaves and vegetables during the drying process [36, 38]. Cellular disruption, porous structure formation, and shrinkage can influence the water uptake capacity and reconstitution to the initial structure of fresh leaves [15, 38]. Rehydration capacity is the ability of the dried

Fig. 5 Rehydration experimental and modelling kinetics



material to absorb water during the rehydration process. In the literature, this complex process is mainly influenced by drying pretreatments, drying methods, morphological structure, chemical composition, immersion media, and rehydration temperature and time [50]. The variation in dry matter observed in our work could influence the rehydration process, but its effect can be neglected, compared to the previous ones, and because of the low moisture values if any remains. More research can be conducted to understand more about this point.

The empirical models used for the analysis of rehydration are presented with experimental data. Apparently, most models have a good fitting with the experimental data, except Exponential related equation and First-order kinetic model. This observation was confirmed with calculation of coefficient of determination and errors evaluation (Table 7). Exponential related and First-order kinetic models had the less R^2 and high values of RMSE, SSE, and X^2, compared with the rest of models. Vega-Gálvez proposed model can be presented as the best fitted model due to high R^2 (0.994) and the least SSE (0.0196). Thus, the model of Vega-Gálvez can used to describe the rehydration kinetics of dried molokhia leaves.

4.6 Rheology

Likewise, for okra and opuntia fruits, molokhia leaves are part of the vegetables and fruits consumed which contain a significant content of mucilage [51, 52]. These vegetables can release mucilage during their various treatments; such as cleaning, washing, chopping, cooking [53].

Due to the limited harvest season and in order to increase the shelf life, molokhia leaves are mostly consumed as dried vegetables throughout the year [15]. In order to observe and follow the liberation of mucilage during time, ground and whole dried molokhia leaves (at 60 °C) were soaked in static water at 50 °C. The dry matter and rheological analysis of the obtained aqueous extract were analysed. The dry matter of the extract partly explains

Table 7 Rehydration fittings result for 60 °C dried molokhia leaves

Model name	R^2	SSE	RMSE	X^2	Model constants
Peleg	0.9938	0.0215	0.0555	0.0031	a = 5.6018, b = 0.48003
First-order kinetic	0.9681	0.1113	0.1180	0.0139	a = 0.038281
Exponential related equation	0.9681	0.1113	0.1180	0.0139	a = 0.038281
Exponential model	0.9941	0.0207	0.0544	0.0030	a = 6.6872, k = − 0.88855
Weibull	0.9935	0.0228	0.0571	0.0033	a = 0.38901, b = 14.3557
Vega-Gálvez model	0.9944	0.0196	0.0571	0.0033	a = 2.1215, b = 5.04, k = 0.78397

Table 8 Rheological properties and dry matter of water extract molokhia leaves (whole and ground leaves)

	Time (h)	Dry matter of extracts %	Apparent viscosity mPa.s	K (Consistency index mPa.s^n)	n (Flow exponent)	R^2	RMSE
Ground leaves	0.5	0.260 ± 0.008	1.939 ± 0.367^c	0.425 ± 0.027^d	1.350 ± 0.047^a	0.945	0.015
	1	0.251 ± 0.035	1.933 ± 0.434^c	1.022 ± 0.298^{cd}	1.152 ± 0.085^{abc}	0.958	0.012
	2	0.272 ± 0.043	3.208 ± 0.762^{ab}	1.203 ± 1.161^{cd}	1.287 ± 0.168^a	0.985	0.013
	3	0.286 ± 0.006	3.923 ± 0.258^a	1.342 ± 0.455^{cd}	1.256 ± 0.073^{ab}	0.982	0.017
	4	0.280 ± 0.010	3.502 ± 0.565^a	3.049 ± 1.881^{bc}	1.067 ± 0.149^{bc}	0.981	0.015
	5	0.287 ± 0.006	3.047 ± 0.553^{ab}	3.672 ± 1.298^{ab}	0.963 ± 0.067^{cd}	0.956	0.018
	6	0.261 ± 0.014	3.152 ± 0.227^{ab}	2.953 ± 0.560^{bc}	1.017 ± 0.044^{cd}	0.976	0.015
	12	0.272 ± 0.001	2.258 ± 0.437^{bc}	2.887 ± 0.588^{bc}	0.945 ± 0.021^{cd}	0.961	0.012
	16	0.256 ± 0.014	2.846 ± 0.227^{abc}	5.510 ± 0.497^a	0.848 ± 0.037^d	0.965	0.013
Whole leaves	0.5	0.135 ± 0.015	0.648 ± 0.039^c	0.211 ± 0.025*	1.253 ± 0.021^a	0.819	0.009
	1	0.210 ± 0.039	0.677 ± 0.032^{bc}	0.237 ± 0.078*	1.255 ± 0.066^a	0.801	0.011
	2	0.206 ± 0.094	0.735 ± 0.108^{abc}	0.658 ± 0.332*	1.056 ± 0.076^{bc}	0.864	0.009
	3	0.235 ± 0.002	0.758 ± 0.022^{abc}	0.357 ± 0.003*	1.17 ± 0.000^{abc}	0.804	0.012
	4	0.244 ± 0.005	0.754 ± 0.043^{abc}	0.421 ± 0.073*	1.14 ± 0.023^{abc}	0.824	0.011
	5	0.224 ± 0.003	0.867 ± 0.128^a	0.654 ± 0.229*	1.092 ± 0.048^{abc}	0.901	0.010
	6	0.239 ± 0.000	0.787 ± 0.038^{abc}	0.334 ± 0.021*	1.207 ± 0.021^{ab}	0.768	0.015
	12	0.240 ± 0.004	0.811 ± 0.040^{abc}	0.503 ± 0.002*	1.119 ± 0.009^{abc}	0.900	0.008
	16	0.216 ± 0.002	0.836 ± 0.100^{ab}	0.798 ± 0.295*	1.026 ± 0.044^c	0.878	0.009

Values followed by the same letter (s),* are not significantly different at $p \leq 0.05$ according to the Tukey's test

the content of water-soluble molecules, including hydrocolloids such as mucilage. As shown in the Table 8, the dry matter of whole leaves after 30 min of extraction had the lowest values and gradually increased to stabilize after several hours. Meanwhile, the ground leaves had a high dry matter compared to whole leaves for all extraction duration. The high dry matter can be related to the high extraction of water-soluble components including hydrocolloids. These results can be confirmed by the rheological behaviour of extracts of both whole and ground leaves.

At the different duration of extraction and for both leaves, the rheological results are shown in the Table 8. The shear stress-shear rate obtained curves were fitted using the Power-law model. In general, whole leaves expressed an apparent viscosity between 0.648 and 0.867 mPa.s, and the lowest values were obtained in the first hour (0.5 h = 0.648 mPa.s, 1 h = 0.677 mPa.s). This result can be associated to the low extraction of mucilage during the first hour as it was seen in dry matter. However, the ground leaves gave an apparent viscosity many times higher than the whole leaves, which ranged between 1.933 and 3.923 mPa.s. The viscosity and also consistency index show clearly a gradual significant increase during extraction time. Apparently, the grinding of leaves facilitated the diffusion of the macromolecules from leaves particles. Also, the lasted extraction gave those water-soluble macromolecules more time to diffuse into the aqueous solution.

The flow exponent (n) greater than 1 shows the thickening behaviour (dilatant) of fluids. Dilatant fluids act as a shear thickening agents when agitated [54]. Whole leaves showed an almost constant flow exponent; however, ground leaves showed a significant gradual decrease from 1.350 to 0.848. This decrease explains a transfer of the dilating behaviour of the aqueous extracts to a pseudoplastic behaviour ($n < 1$).

Unlike dilating fluids, pseudoplastic fluids become thinner when the shear rate increases. The elements contained in the pseudoplastic fluids follow the direction of the flow, so deformation and breakdown of the aggregates occur whereby the viscosity is limited [54]. The diffusion of hydrocolloids from the plant material to the immersion solution increases their concentration and thus affects the rheological behaviour of solutions. It appears that the longer the extraction time, the higher the hydrocolloid extraction rate, and hence a high consistency index and low flow exponent. This finding is in agreement with that of Koocheki et al. [55], who found that increasing of hydrocolloids concentration had increased the consistency index and decreased the flow behaviour of the prepared solutions.

5 Conclusion

The analysis done on fresh molokhia leaves exhibited its considerable content of bioactive compounds, such as phenolics and flavonoids, thereby their importance to reduce the effect of oxidative compounds. This functional property and others could justify the uses of this leafy vegetable in culinary and medicinal preparation. Processing techniques such as drying can affect the quality and biochemical composition, accordingly its biological activities. In the present work, shade drying was the most effective to preserve leaf colour, composition, and antioxidant activity. In contrast, convective drying at 80 °C had the worst effect on leaves quality and composition. Many used empirical models have demonstrated a good fitting with drying kinetics ($R^2 > 0.980$). Meanwhile, rehydration kinetics had the best fitting with the Vega-Gálvez model. All those models had the ability to well describe the kinetics. The state of leaves (ground, whole) affects significantly the diffusion of hydro-soluble compounds and then the rheological properties of leaves prepared extract. The processing of leaves should take into consideration the effecting factors such as temperature, power input, and others in order to produce a high-quality product with more functional activities preservation.

Authors' contributions Conceptualization, MG, BF and IK; methodology, MG, BF and IK; validation, MG and BF; formal analysis, MG and BF; data curation MG and BF; writing—original draft preparation, MG; writing—review and editing, MG; project administration, MG and IK. All authors read and approved the final manuscript.

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Data availability The datasets generated during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests The authors declare no conflicts of interest.

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