Microwave-drying kinetics of lemon (*Citrus limon*) determining some quality attributes of dried product

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Abstract: Drying kinetics of lemon fruits undergoing microwave-drying was investigated, furthermore, mathematical drying models were statistically analyzed to simulate drying kinetics fitted to the obtained experimental moisture ratio results. The investigation was conducted under three levels of microwave power of 550, 770 and 1100 W. The variations in lemon moisture content (M.C.) were recorded versus time until reaching to 14.28%, 12.99% and 11.73% d.b. and 14.15%, 11.60% and 12.86% d.b. for whole fruit without and with 1% NaOH pretreatment, respectively, at previously power levels, respectively. Meanwhile, for slices, the M.C. (at the same previously power levels, respectively) reached (12.86%, 14.15% and 15.47% d.b.), (16.82%, 12.86%, and 14.15% d.b.) and (12.86%, 14.15%, and 15.47% d.b.) for thicknesses of 4, 5 and 6 mm, respectively. The whole lemon fruit with preated-1% NaOH recorded highest percentages of: total crude fats (1.782%), total soluble sugar (6.88%), citric acid (6.62%) and ascorbic acid (46.05 mg/100g) compared to other treatments.

Keywords: drying kinetics; microwave-drying; lemon; moisture ratio; NaOH pretreatment; quality attributes.

Citation: Radwan, S. E. N., M. M. Hassan, and T. H. Ahmed. 2023. Microwave-drying kinetics of lemon (*Citrus limon*) determining some quality attributes of dried product. Agricultural Engineering International: CIGR Journal, 25(1): 211-224.

1 Introduction

Lemon (*Citrus limon*) is nutritious fruit with a myriad of health benefits. It boasts one of the nature's highest Vitamin C concentrations, total phenolic content (TPC) and a unique flavor and aroma (Santos and Silva, 2008). Furthermore, it contains other nutrients with antioxidant properties such as; phenylpropanoids and flavonoids which strengthen the immune system, prevent the development of cancer cells, and prevent free radicals from damaging body tissues (Benavente-Garc **´n** and Castillo, 2008).

In Egypt, lemon is grown along the banks of the River Nile in four areas: Delta, New lands, Upper

Received date: 2022-01-13 **Accepted date:** 2022-12-26

Egypt, and Middle Egypt and has always played an important social and economic role in people's lives. In year 2018, about 2002567 tonnes of gross production of lemons and limes is estimated (FAOSTAT, 2018). These large amounts of lemon production surly need to right methods of handling and preserving.

As drying process compared to others remains a promising method of food preserving technology (Abd Allah and Ahmed, 2020). The benefit of drying fruits and vegetables is not only to preserve crops as food, but also reduce package and transport cost in terms of weight and volume, while offering the possibility of adding value to harvested commodities. Lemons contain 90% of water. Thus, high water content causes the growth of microbial which eventually shortens the overall shelf life of the lemons. Therefore, drying works as a preservation method by removing the water from the lemons in order to extend its shelf life. Thus far, there are various drying methods which have been used for drying of slices such as closed type solar drying,

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March, 2023

infrared drying, open sun drying, and hot air drying (Chen et al., 2005; Swanson, 2009). Moursy et al. (2014) studied the effect of hot-air drying temperature on vitamin C content of whole lemon fruit and fruit cut into halves, quarters and slices. The optimum temperatures were 90 $^{\circ}$ C for samples cut into halves and 100 °C for samples cut into quarters and slices. Lee et al. (2015) studied drying kinetics of lemon slices with 3 mm thickness and determined dried product quality using heat pump and oven. The highest Vitamin C and total phenolic content (TPC) were 6.74 mg AA / g dry weight and 13.76 mg GA / g dry weight, respectively, for oven dried lemon slices at 50 °C. Moreover, Papoutsis et al. (2017) studied the effect of freeze drying, hot air and vacuum drying on dried lemon pomace polyphenols and antioxidant capacity at 70 $^{\circ}$ C, 90 ℃ and 110 ℃.

Implementing microwave radiation in drying processes causes internal faster heating compared to other drying equipment. This heating method is advantageous in thermal drying, since favorable properties of water and other polar liquids generate heat in the wet parts of the drying material, and hence extracting the moisture. This method considerably reduces drying time and promotes the quality of the dried sample (Mirzabeigi Kesbi et al., 2016).

Recently, researchers have studied drying behavior assisted-microwave of various natural materials such as: microwave-assisted infrared drying of lentil seeds (Heydari et al., 2020); microwave freeze drying of button mushroom (Duan et al., 2021); microwave drying of banana blossoms (Jha et al., 2021); microwave heating of pineapple juice (Chua and Leong, 2020).

In spite of the appreciable number of literature studied the drying of various agro-products, limited studies on lemon drying, particularly microwavedrying, have been conducted and have not been noted this issue considerably. In this context, Tekgül and Baysal (2019) evaluated a combined vacuum microwave drying (VMD) conditions of lemon peel using response surface methodology (RSM) and to observation of its effects on the quality and volatile compounds. The optimum vacuum power and temperature were 584 watt and 50°C, respectively. Drying significantly affected the volatile compounds, antioxidant activity, total carotenoid, and ascorbic acid contents of lemon peel powders. Mirzabeigi Kesbi et al. (2016) investigated a combined microwave-convective drying behavior of lemon slices with specific powers of 0.97 and 2.04 W g⁻¹ assisted with 50°C, 55°C and 60°C inlet air temperatures.

Although applying microwave have been evaluated combinable with either convective or vacuum for drying application of lemon slices and lemon peels, there are no researches noticed in the literature focusing on solely microwave-drying for lemon fruit undergoing NaOH pretreating conditions within assessing the quality attributes of dried lemon. Therefore, this study aimed to explore the microwave-drying kinetics of NaOH pretreated lemon fruit and differed-thickness lemon slices based on mathematical drying models at different microwave power levels, and evaluate the effect of applied microwave-drying on moisture ratio, drying kinetics, drying efficiency, the specific energy consumption of lemon as well as quality attributes of dried lemon i.e. vitamin C, total soluble sugar, and total crude fat.

2 Materials and methods

2.1 Sample preparation

Freshly-harvested lemon fruits (*Citrus limon*) were picked in an advanced stage of ripeness. The initial moisture contents of the harvested lemon were 84.53% in whole lemon fruit and 85.45% in lemon slices.

2.2 Pretreatment of the sample

Since the whole fruit lemons are a complex product with an outer waxy cuticle and pulpy material inside, the outer waxy cuticle affects the moisture diffusion from the lemons during the drying process. A chemical pre-treatment is generally applied to decrease the skin resistance and hence improving moisture diffusion through the waxy cuticle, for this experimental work the samples of the whole lemon were blanched by dipping in a boiling solution containing 1% of NaOH for 1.5 min and immediately cooled by immersing in running cold water as recommended for grapes by Pangavhane et al. (1999) and Doymaz and Pala (2002).

2.3 Drying equipment

Microwave oven is an electromechanical appliance that is used for cooking or heating food through insulating heating. This oven uses microwave radiation, as it warms food only unlike conventional ovens, to heat water and other similar molecules that are polarized within the food. Here, heating is evenly between the inside and outside of the food due to the penetration of microwave radiation inside the food, model R-340 R (S). 230-240 V, 50 HZ, input 1.60 kW, 7.2 A, maximum output 1100 W (IEC), and frequency 2450 MHz.

2.4 Drying procedures

The main experiment was carried out to investigate using a domestic microwave-drying as a drying equipment of lemon fruit in order to evaluate the performance of microwave-drying method for drying two different forms of lemon fruit (whole and slices). The experimental parameters (as shown in Figure 1) included: three different levels of microwave power (550, 770, and 1100 W); two forms of lemon samples (whole fruit (with and without pretreatment by 1% NaOH) and slices with three thicknesses (4, 5, and 6 mm). The lemon samples were regularly spread on the microwave dryer. The weight of samples at the beginning of the experiment was 100 g. The weight was measured every 1 minute. The experiments were conducted till the moisture content reached to 11.73%-16.82% (d.b).





d = Dry sample mass (g).

2.5.2 Moisture ratio and mathematical modeling

Moisture ratio (*MR*) removal was calculated as following:

$$\mathbf{MR} = \frac{\mathbf{M}_t \cdot \mathbf{M}_e}{\mathbf{M}_i \cdot \mathbf{M}_e} \tag{2}$$

where,

MR = Moisture ratio of the sample (%),

 M_t = Moisture content at any time of drying (kg_{water}/kg_{dm}),

 M_i = Initial moisture content (kg_{water}/kg_{dm}),

 M_e = Equilibrium moisture content (kg_{water}/kg_{dm}).

Four semi-empirical models were applied to fit the

2.5 Drying kinetics

2.5.1 Moisture content

Moisture content (M.C.) removal was set by drying the samples till reaching a fixed weight. Moisture content of lemon types were 85.4% and 84.5% w.b. for slices and whole lemon, respectively. The Moisture content was calculated as following:

M.c. (w.b.) (%)
$$= \frac{W \cdot d}{W} \times 100$$
 (1)

where,

M.C. (w.b.) (%) = Moisture content of the sample (%),

W = Wet sample mass (g),

experimental moisture data because they are widely

used in drying agriculture products, as listed in Table 1.

Table 1 Mathematical models given by various authors for the drying curves

No	Model Name	Model	References			
1	Newton	MR = exp(-kt)	O'Callaghan et al., 1971; Liu and Bakker-Arkema, 1997			
2	Page	$MR = exp(-kt^n)$	Agrawal and Singh, 1977; Zhang and Litchfield, 1991			
3	Modified Page	$MR = exp[-(kt)^n]$	Agrawal and Singh, 1977; Zhang and Litchfield, 1991			
4	Henderson and Pabis	$MR = a \ exp(-kt)$	Westerman et al., 1973; Chhinnan, 1984			
where,		The specifi	c energy (Qs, MJ kg _{water} ⁻¹) was			

where,

 $k = Drying constants, (min^{-1}),$

t = Drying time, (min),

a = Drying constant, (dimensionless),

n = the number of constants.

Regression analyses were done by using the statistical models. The coefficient of correlation (r) was one of the primary criteria for selecting the best equation to define the thin layer drying curves of lemon samples (O'Callaghan et al., 1971). In addition to r, the various statistical parameters such as; reduced chisquare (χ^2) , mean bias error (*MBE*), and root mean square error (RMSE) were used to determine the quality of the fit model. These parameters can be calculated as follows:

$$X^{2} = \frac{\sum_{i=1}^{N} (MR_{obs,i} - MR_{calc,i})^{2}}{N - n}$$
(3)

$$MBE = \frac{1}{N} \sum_{i=1}^{N} (MR_{obs.,i} - MR_{calc.,i})$$
(4)

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} (MR_{obs,i} - MR_{calc,i})^{2}\right]^{\frac{1}{2}}$$
(5)

where.

 $MR_{obs,i}$ = stands for the observed moisture ratio found in any measurement,

 $MR_{calc.,i}$ = stands for the observed moisture ratio found in any measurement,

N, and n = are the number of observations and constants, respectively (Pangavhane et al., 2002).

2.6 Specific energy and drying efficiency

Energy consumption in drying (Q_t) was calculated as follows (Darvishi, 2012):

$$Q_t (W.min) = P \times t$$
 (6)

where,

$$Q_t$$
 = Energy consumption in drying (W min),

P = Required power (W),

T = Drying time (min).

calculated as the energy needed to evaporate a unit mass of water (Mousa and Farid, 2002; Soysal et al., 2006).

$$\mathbf{Q}_{s} \left(\mathbf{MJ}/\mathbf{kg}_{water} \right) = \frac{\mathbf{Q}_{s} \times 60}{m_{w} \times 1000}$$
(7)

where,

 Q_s = Specific energy consumption in drying (MJ kg_{water}⁻¹),

 Q_t = Energy consumption in drying (W min),

 m_w = Mass of evaporated water (g).

The drying efficiency $(\eta, \%)$ was calculated as the ratio of the heat energy utilized for evaporating water from the sample to the heat supplied (Yongsawatdigul and Gunasekaran, 1996; Soysal, 2004).

$$\eta (\%) = \frac{m_w \times \lambda_w}{Q_t \times 60} \times 100$$
(8)

where,

 η = Drying efficiency (%),

 m_w = Mass of evaporated water (g),

 λw = Latent heat of vaporization of water (kJ kg⁻¹),

 Q_t = Energy consumption in drying (W min).

The latent heat of vaporization of water at the evaporating temperature of 100°C was taken as 2257 kJ kg⁻¹ (Hayes, 1987).

2.7 Quality attributes

2.7.1 Total crude fat

Total crude fat was estimated as follows; 0.5 g of each powdered sample was extracted using a continuous extraction apparatus (Soxhlet) with solvent petroleum ether (b.p 60 $^{\circ}$ C -80 $^{\circ}$ C) for 16 hours. Each extract was dried over anhydrous Na2SO4 and evaporated to dryness. The residue was dried at $80 \,^{\circ}$ for 10 minutes; cooled; weighed and expressed as lipids %. (AOAC, 1984).

2.7.2 Total soluble sugar

Total soluble sugars were determined by the anthron methods (Sadasivam and Manickam, 1996) through adding 3 mL, anthron reagent to 0.1 ml filtrate, then heated for 10 min in a boiling water bath, cool

rapidly and the developed green color was read at 630 nm by spectrophotometer.



(b) without NaOH-pretreatment Figure 2 Effect of microwave power on whole M.C. lemon fruit' VS drying time



Figure 3 Effect of microwave power on M.C. lemon slices' VS drying time at different thickness

Table 2 Values of chi-square (χ	²), mean bias error (MBE)) and root mean square error	(RMSE) for all models	of microwave, at

microwave power (550 W)

		Lemon samples					
The model			Slices thickness (mm)		Whole fruit		
		4	5	6	Without pre-treatment	With pre-treatment	
	χ^2	0.001763	0.004916	0.018859	0.018859	0.014675	
Newton	MBE	0.030156	0.053667	0.076301	0.081751	0.069398	
moder	RMSE	0.041107	0.068575	0.132673	0.132673	0.117294	
Henderson model	χ^2	0.000836	0.003079	0.018167	0.018167	0.017266	
	MBE	-0.002910	-0.009454	-0.018933	-0.018933	-0.028514	
	RMSE	0.027675	0.053025	0.125477	0.125477	0.122913	
Page model	χ^2	0.100307	0.000126	0.012923	0.012923	0.091214	
	MBE	-0.216886	0.002895	0.028683	0.028683	-0.186722	
	RMSE	0.303230	0.010723	0.105832	0.105832	0.282511	
Modified page model	χ^2	0.000035	0.024926	0.012888	0.166735	0.048213	
	MBE	0.000167	0.015913	0.028401	0.161734	-0.151175	
	RMSE	0.005636	0.150860	0.105688	0.380136	0.205393	

2.7.3 Citric acid

The citric acid in the lemon was determined according to the method reported in AOAC (1975) by titration with 0-1 N sodium hydroxide after adding a few drops of phenolphthalein as an indicator.

2.7.4. Ascorbic acid (Vitamin C)

The ascorbic acid in the dried lemon was determined according to the method reported in AOAC (1975) by titration with dichlorophenol indophenol blue dye.

3 Results and discussion

The discussion of the obtained results was under the following heads:

3.1 Drying kinetics of moisture content versus drying time at different microwave power levels

Generally, the results revealed that, increasing power from 550 to 1100 W caused reduction in draying time of lemon. Thus, the obtained results were conducted under three different levels of abilities (550, 770, and 1100 W). The changes in lemon M.C. were recorded versus time until reaching a moisture content of 10.5%-12.5% (d.b) approximately as a level similar to that of the commercial sample of dried samples.

Figures 2 and 3 show the difference in M.C. versus drying time for lemon samples at 550, 770, and 1100 W, with different lemon samples slices thicknesses, (4, 5, and 6 mm), whole fruit and whole fruit with NaOH-

pretreatment. The results indicated that, M.C. of whole fruit decreased to 14.28% (d.b.) after 15 min at power 550 W, to 12.99% (d.b.) after 10 min at 770 W, and to 11.73% (d.b.) after 7 min at power 1100 W. While soaking in (1% NaOH), M.C. decreased to 14.15% (d.b.) after 16 min at 550 W, to 11.60% (d.b.) after 14 min at 770 W and to 12.86% (d.b.) after 9 min at 1100 W. Furthermore, the results indicated that M.C. of lemon slices (4 mm of thickness) decreased to 12.86% (d.b.) after 24 min at 550 W, to 14.15% (d.b.) after 11 min at 770 W and to 15.47% (d.b.) after 10 min at power 1100 W. Meanwhile lemon slices (5mm of thickness), M.C. decreased to 16.82% (d.b.) after 23 min at 550 W, to 12.86 % (d.b.) after 11 at 770 W min and to 14.15% (d.b.) after 11 min at power 1100. While lemon slices (6 mm of thickness), M.C. decreased to 12.86% (d.b.) after 13 min at 550 W, while to 14.15% (d.b.) after 10 min at 770 W, and to 15.47% (d.b.) after 6 min at power 1100 W.

3.2 Mathematical modeling of microwave-drying of lemon

The data of M.C. versus time were transformed to moisture ratio versus time to normalize the drying curves. The data at different curve fitting computations with the drying time were conducted on four drying models predestined by the previous works. The results of statistical analyses pledge on these models are given in Tables 2-4, respectively. The predestined of the models were based on R^2 , χ^2 , *MBA* and *RMSE*. For different thickness-lemon slices at power levels of 550, 770, and 1100 W, the Newton and Henderson and Pabis's models were the best descriptive models for all lemon samples this due to the higher values of coefficient of determination R^2 (0.97099 to 0.97312).

The Newton drying constant (k) of lemon slices and whole lemon fruit with pre- treatment increased with the increase of microwave power but the drying constant (k) of whole lemon fruit without pre-treatment decreased with the increase of microwave power. The drying constant (k) of Henderson and Pabis's model increased with the increase of microwave power. - The drying constant (kp) of Page's model increased with the increase of microwave power. Although the 5 mm slides give valuables unsteady. The Values of drying constant (k) of Modified Page's model are unsteady. - Both studied models could describe the drying behavior of whole lemon fruits and slices satisfactorily. However, the Newton's model and Henderson and Pabis's considered more proper for describing the drying behavior and predicting the changes in moisture content of both forms of lemons.

Table 3 Values of chi-square (χ^2), mean bias error (MBE) and root mean square error (RMSE) for all models of microwave, at microwave power (770 W)

		Lemon samples					
The model			Slices thickness (m	ım)	Whole fruit		
		4	5	6	Without pre-treatment	With pre-treatment	
	χ^2	0.000260	0.002020	0.008194	0.006325	0.028084	
Newton model	MBE	0.008222	-0.033259	0.054574	0.041876	0.117956	
	RMSE	0.015361	0.042855	0.085877	0.075447	0.1614855	
	χ^2	0.000168	0.003626	0.010350	0.009322	0.012669	
Henderson model	MBE	0.000591	0.013084	-0.018401	-0.018031	0.038933	
	RMSE	0.011720	0.054471	0.090995	0.086359	0.104209	
	χ^2	0.000060	0.000416	0.003109	0.003541	0.033382	
Page model	MBE	-0.000826	-0.003764	0.006875	0.004974	-0.137918	
	RMSE	0.007035	0.018443	0.049868	0.035881	0.028613	
	χ^2	0.000369	0.000418	0.003090	0.003816	0.033378	
Modified page model	MBE	-0.015963	-0.003874	0.006399	0.010359	-0.191981	
model	RMSE	0.018217	0.018484	0.049717	0.055250	0.307131	

Table 4 Values of chi-square (χ^2), mean bias error (MBE) and root mean square error (RMSE) for all models of microwave, at

microwave power (1100 W)

The model		Lemon samples				
		Slices thickness, (mm)			Whole fruit	
		4	5	6	Without pre-treatment	With pre-treatment
	χ^2	0.002185	0.019975	0.022619	0.005982	0.013813
Newton model	MBE	0.020968	-0.096166	0.095747	0.011987	0.076488
	RMSE	0.0443471	0.134756	0.137293	0.071606	0.1108057
	χ^2	0.335820	0.025099	0.028944	0.022440	0.010263
Henderson model	MBE	0.317948	0.022108	-0.029473	0.069651	-0.011344
	RMSE	0.546358	0.143302	0.138910	0.142830	0.089344
Paga modal	χ^2	0.077348	0.003041	0.009911	0.006230	0.032729
rage model	MBE	-0.194159	-0.007765	0.077337	0.011530	-0.145477

	RMSE	0.248753	0.049881	0.081285	0.066706	0.170564
	χ^2	0.077032	0.003058	0.000024	0.006245	0.037407
Modified page model	MBE	-0.269427	-0.008184	0.001624	0.011732	-0.251032
	RMSE	0.380422	0.050024	0.003978	0.066787	0.377106

3.3 Effect of studied parameters on specific energy and drying efficiency

Specific energy and drying efficiency of lemon samples under experimental drying conditions were shown in Figures 4 and 5. Data clarified that, whenever the consumed specific energy for drying lemon was low and thus, the drying efficiency increased. For 100 g of lemon samples the lowest specific energy value was 5.50 MJ kg⁻¹ for 6 mm slices at 1100 W and thus achieved the highest drying energy efficiency 41.04%. The highest value of specific energy was 10.7 of 4 mm slices at 550 W, and therefore its drying energy efficiency was low 21.88%. The reason for the high or low values is due to the shortening of the drying time.



(b) whole lemon fruits with/ without NaOH pretreatment Figure 4 Specific energy VS microwave power



(b) whole lemon fruits with/ without NaOH pretreatment Figure 5 Drying efficiency VS microwave power

3.4 Quality attributes of microwaved-dried lemon at different power levels

Figure 6 illustrated the changes in total crude fats of sliced lemon, whole fruit with 1% NaOH pretreatment and without and at different levels of microwave power. The percentage of total crude fats of microwave ranged

from 1.628% to 1.751% for the whole lemon, from 1.244% to 1.483%, for sliced lemon and from 1.659% to 1.782% for whole fruit with pretreatment. Total crude fats concentration increased with the increase of microwave power. The whole lemon fruit with 1%

NaOH pretreatment recorded the highest percentages of

total crude fats comparing to other dried samples.



Figure 7 illustrated the changes in total soluble sugar as related to microwave power of sliced lemon, whole fruit with 1% NaOH pretreatment and without at different levels of microwave power. The total soluble sugar was ranged from 6.48% to 6.79 % for the whole lemon samples, from 5.47% to 6.09% for the sliced

lemons and from 6.56% to 6.88% for whole lemon fruit with pretreatment. These results revealed that, the whole lemon fruit with 1% NaOH pretreatment recorded the highest percentages of total soluble sugar comparing to other dried samples



Microwave samples

Figure 7 Total soluble sugar at different power levels for whole and sliced microwaved-dried lemon fruits

Figure 8 presented the percentage of citric acid in sliced dried lemons, whole lemon fruit with 1% NaOH pretreatment and without as related to microwave power. The dried whole lemon fruit with 1% NaOH

pretreatment showed the highest citric acid percentages for all treatments comparing to other samples dried. The recorded percentages of citric acid ranged from 6.18% to 6.62%, from 6.07% to 6.51% for whole fruit without

to 5.52%.

pretreatment. However, for the dried lemon slices the corresponding values of citric acid ranged from 4.72%



Figure 9 presented the changes in Ascorbic acid (mg/100g) as related to different levels of microwave power for sliced dried lemons, whole lemon fruit with 1% NaOH pretreatment and without. The ascorbic acid in the whole lemon treatment ranged from (44.57 to $45.74 \text{ mg}/100 \text{ g}_{dry \text{ matter}}$), the corresponding values for

the sliced lemons ranged from (40.61 to 43.05 mg/100 $g_{dry matter}$) and from (44.86 to 46.05 mg/100 $g_{dry matter}$) for whole lemon fruit with pretreatment. It is obviously that the higher values of ascorbic acid were achieved with the pretreatment of whole fruit with 1% NaOH pretreatment.



Figure 9 Ascorbic acid at different power levels for whole and sliced microwaved-dried lemon fruits

4 Conclusions

The reduction in moisture content of whole fruit of lemon and lemon slices was varied with the experimental treatments. It was increased with the increase of microwave power. The microwaved-dried whole lemon with 1% NaOH showed the highest values of quality attributes of total crude fats, total soluble sugar, citric acid, and ascorbic acid (vitamin C) percentages' comparing to other samples. The microwave power of 1100 W recorded the highest percentage of: total crude fats (1.782%); total soluble sugar (6.88%); citric acid (6.62%); and vitamin C (64.05 mg/100 $g_{dry\ matter})$ for whole lemon fruit with 1% NaOH pretreatment. Thus, the best treatments that achieved the highest level in quality characteristics are for the whole fruit with 1% NaOH-pretreated in the Additionally, microwave oven. Newton's and Henderson and Pabis's models considered more proper mathematical models for describing the microwavedrying behavior and predicting the changes in moisture content of all treatments of sliced and whole fruit of lemons. The drying efficiency of microwave is the highest at lemon slices thicknesses of 6 mm due to the low consumption of specific energy.

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