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Drying of Madder (*Rubia tinctorum* L.) with Microwave and Determination of Dyestuff

Muhammed BIYIKLI [1], Hürkan Tayfun VAROL[2*]

[1]Department of Field Crops, Isparta University of Applied Sciences, Faculty of Agriculture Isparta, Turkey
[2]Department of Agricultural Machinery and Technologies Engineering, Isparta University of Applied Sciences, Faculty of Agriculture Isparta, Turkey
*Corresponded author e mail: hurkanvarol@isparta.edu.tr, Phone: 05346977984

Abstract: The objective of this study was to determine the effects of microwave power densities on the moisture ratio, drying rate, color parameters, energy consumption and total dye content of madder (*Rubia tinctorum* L.) plant at different microwave power. For this purpose, madder with the beginning moisture content of 80.84% ± 0.8 were dried using microwave until moisture content reach to average of 4.67% ± 1.92 (dry basis). Microwave trials at 180 W ,360 W, 540 W, 720 W and 900 W lasted for 41, 18, 10, 8 and 7 min respectively. Addition to the trails' madder was dried with oven at 105 °C for 24h for comparison. Determining the most appropriate drying models were selected considering the smallest RMSE along with 13 different drying model. After drying trials samples were grounded and color analysis was performed. Energy consumption values were determined and converted into specific energy consumption values and the most efficient application was obtained at 540 W with a value of 3.128 Whg^{-1}. When all treatments were compared in terms of total dye content, statistically significant differences were found ($p<0.01$). Total dye content observed at the highest level of 2.32% in 180 W treatment. Therefore, drying recommended at 180 W due to major property of madder was total dye content.

Keywords: Agricultural drying, Total dye content, Energy consumption, Drying modelling, color parameters

Introduction

Many different disciplines have focused on the theme of "Ecological Product" with the increasing orientation towards the theme of conscious consumer worldwide. Recently, in the face of the negativity towards the use of synthetic products, the threat to both environmental health and human health has encouraged the use of natural and sustainable raw materials. Some synthetic dyes have been reported to have mutation accelerating [30,35], genotoxic [39], carcinogenic, dysfunctional, sperm motility inhibiting effects [9, 16, 22]. These synthetic dyes can cause allergies, toxic wastes and damage to the human body [38]. Natural dyes have no harmful effects on the environment because they are renewable and natural. Therefore, they are healthier and more attractive for consumers.

One of the most important dye plants and the plant from which the color "Turkish Red" is obtained is a very valuable member of the Rubiaceae family [15]. Due to the anthraquinone pigments found in its roots and rhizomes, it has pioneered the transformation of textiles symbolizing the nobility of royal families and carpets decorated with motifs reminiscent of ancient cave paintings into magnificent works of art and has witnessed the secret of red throughout history [19].

The use of this plant throughout history and its place in the world economy is due to its use as a source of dyestuffs. It is located in the springs where madder was grown in Malatya at the end of the 19th century [8]. Madder has met the needs of foreign dyer as well as the needs of local dyers. It was reported that in the early 19th century, a significant amount of madder was exported from Ereğli to Aleppo, from Gördes to Izmir dye houses and to foreign markets. Madder was also exported to England, Austria, America, Russia and Rumelia from Izmir [8]. Although it lost its importance with the discovery of synthetic dyes in the second half of the 19th century, it has increased its popularity as the subject of new research and projects and has gained a new reputation in many fields.

Due to the presence of alkaloids, phenols, cardiac glycosides, flavonoids, terpenes, tannins, coumarins and essential oils in its structure [33], the madder plant has been reported to have many biological activities such as anticancer, anti-inflammatory, diuretic, vascular relaxant [33], antiarthritic, antifungal, antibacterial, antiviral, antiplatelet and neuroprotective [18]. Due to these properties, it is used in many industries such as medicine, health, cosmetics, agriculture and food [23]. In a study on the wound-healing effect of the madder plant, it was emphasized that the ointment with madder extract showed wound healing effect in mice [28]. Madder, which is the subject of various ethnobotanical researches, has been proven to be effective in the treatment of disorders such as blood pressure, heart diseases, kidney stones [12], tumors, bladder [18]. It has also been used in the treatment of breast, prostate and colon cancer [23], eczema and fungus, jaundice, sciatica, paralysis, postpartum menstrual regulator effects have been reported [20].

Various projects are currently being carried out in Europe for the cultivation of dye plants, which are the source of vegetable dyes. In Italy, the PrisCA project, which includes the cultivation of *Rubia tinctorum* and the production of dyestuffs, is being carried out. In recent years, Isparta University of Applied Sciences, Department of Field Crops, Süleyman Demirel University Faculty of Fine Arts, Marmara University Textile Engineering, Trakya University Havsa Vocational School, Department of Park and Horticulture, Tokat Gaziosmanpaşa University Natural Dyes Application and Research Center, Diyarbakır GAP UTAEM and some public institutions (Isparta Municipality, Burdur Municipality, Balıkesir-Sındırgı Municipality) are trying to develop the agriculture and industry (textile, chemistry, pharmacy) of madder plant in Turkey [11].

Considering the yield of the madder as 30%, Türkiye's requirement for 11 tonnes of dry rhizomes it is assumed that 36.6 tonnes of fresh rhizomes must be collect based on survey research [3, 10]. Since there is not much information in the literature about the conditions under which this product is stored and dried, traditional drying methods are generally preferred. In this method, it has been found in our survey researches that the rhizomes of madder give more than 70% waste, and in some cases this value reaches up to 75-80% due to microbial infections. Madder is susceptible to rapid deterioration due to its high moisture content. Inadequate drying and storage conditions and high relative humidity environments can trigger degradation by increasing microbial activities in the rhizomes [43]. After harvesting, it is important to preserve the quality of dyestuff with appropriate drying and storage conditions. For this reason, controlling the water activity of the product and preventing microbial degradation by performing this process in a short time will ensure the preservation of the unique color, odor and dye of the madder plant.

The microwave drying process has the advantages of short drying time, low energy consumption and no significant changes in color, scent, and nutritional content. In this method, the water molecules in the sample to be dried are repels by vibration at high frequency using a magnetron. The kinetic energy generated by the vibration of the water molecules is converted into heat energy like this the material is heated globally. This method provides drying in a short time comparing to traditional (shade drying) method by reducing the moisture content of the plant and preventing degradation. In many studies, it is reported that microwave drying technique is applied in drying products with high moisture content such as fruits, vegetables, and medicinal plants [25]. This research was carried out to determine the drying behavior of madder plant under different drying conditions in a laboratory type microwave dryer and the determine of different microwave power densities on the change total dyestuff content in madder.

Material And Method

Material

Dry madder samples were harvested from the trial field of Isparta University of Applied Sciences. The samples were dried right after harvest. Prior to trials, the madder roots were cut uniformly as same sized material. For each experiment 50 gram of madder was used and spread on large plate. Drying experiments were carried out employing a microwave dryer of Arcelik MD 594 model. Power levels of 180, 360, 540, 720 and 900 W were selected. Madder samples were weighed at one-minute intervals using balance, until the reaches to constant weight.

Determination of moisture content

Madder samples of 100 g placed in the container then dried for 24h in a 105 °C oven. Determining moisture crucial for understand the behavior of drying. With the method of AOAC [4], moisture was calculated (Eq 1).

$$\%moisture = \frac{M_1-M_2}{m} * 100 \quad (1)$$

in equation;

M_1; Fresh madder weight+ container weight (g)

M_2; Dried madder weight + container weight (g)

m; Sample weight received (g)

Moisture ratio

The moisture ratio (MR) was determined by considering the moisture content over time (M(t)), along with the beginning moisture content of the samples (M0), and the equilibrium moisture content (Me). Neglecting Me was justified as it significantly lower than both M_0 and Mt. [36].

$$MR = \frac{M(t)-M_e}{M_0-M_e} \quad (2)$$

Drying rate

The drying rate (DR) was found by taking the derivatives of the moisture content versus drying time curves with Equation 3 below.

$$DR = \frac{M_{t+dt}-M_t}{d_t} \quad (3)$$

where: DR, represents the drying rate, Mt+dt: Moisture presence at time t+dt, Nt is the moisture presence at time t, dt is the time while the moisture presence is calculated during the drying [17].

Mathematical modeling

Drying in agricultural field, lots of mathematical models tries to explain the drying behavior of the product. The experimental models used within the scope of the study are given in Table 1. These models are evaluated by comparing their performance criteria's such as root mean square error (RMSE), coefficient of determination (R^2), model efficiency (EF) and chi-squared (χ^2). Equilibrium for R_{MSE} , EF and χ^2 are as follows:

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i}-MR_{pre,i})^2}{N-Z} \quad (4)$$

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

$$EF = \left[\frac{\sum_{i=1}^{N}(MR_{pre,i}-MR_{exp,average})^2-(MR_{pre,i}-MR_{exp,i})^2}{\sum_{i=1}^{N}(MR_{pre,i}-MR_{pre,average})^2}\right] \quad (6)$$

where $MR_{exp,i}$ and $MR_{pre,i}$ are experimental (exp) and predicted (pre) dimensionless MR values $MR_{exp,average}$ and $MR_{pre,average}$ are mean values of exp. and pre. MR values respectively; N is the total number of experiment; and Z is the constant number of mathematical model.

Table 1. Mathematical models used for drying

No	Mathematical models		References
1	Newton	MR=exp(-kt)	[5]
2	Page	MR=exp(-kt^n)	[37]
3	Modified Page	MR=a exp[-(kt^n)]	[51]
4	Logarithmic	MR=a exp(-kt)+c	[51]
5	Two Term	MR=a exp(-k_1t)+b exp(-k_2t)	[1]
6	Verma et al.	MR=a exp(-kt)+(1-a)exp(-gt)	[49]

7	Midilli et al.	MR=a exp(-kt^n)+bt	[33]
8	Henderson and Pabis	MR=a exp(-kt)	[21]
9	Diffusion approximation	MR=a exp(-kt)+(1-a) exp(-kbt)	[2]
10	Jena&Das	MR=a exp(-kt+b√t)+c	[24]
11	Demir et al.	MR=a exp(-kt)^n+c	[14]
12	Kulcu et al.	$MR = \frac{a^b \exp(-kt^n)}{c^t} + d$	[27]

Color Measurement

Color has a significant impact on consumers' perception of product quality [29]. This perception varies with ambient lighting conditions. In the CIE color space, the primary international color parameters are L*, a*, and b*. L* denotes luminosity, while a* and b* represent the green/blue and red/yellow axes, respectively, with negative and positive values. However, these measured L*, a*, and b* values do not directly correspond to consumers' perception of color. To bridge this gap, hue angle (α) and chroma (C) are calculated from these values. The hue angle is derived mathematically from a* and b* in the Lab* color space, ranging from 0° to 360°, and corresponds to specific hues such as red-violet, yellow, bluish-green, and blue. Research indicates that the hue value facilitates a more precise interpretation of the a* and b* values since it represents the angular value of these parameters [49]. Chroma (C), also known as color intensity, measures the vividness of color. It takes into account the levels of redness and yellowness, with higher values indicating vibrant colors and lower values indicating dull colors. Equations for calculating C and α values are provided below [41]. Both CIE's Lab* and Hunter Lab's Lab color spaces are frequently employed in the literature to represent the color of food products. Chroma and hue values were calculated using Equations 7 and 8, respectively.

$$C = \sqrt{(a^2 + b^2)} \tag{7}$$

$$\alpha = tan^{-1}\left(\frac{b}{a}\right) \tag{8}$$

Determining energy consumption

During the trails energy needs for removing the inner water content was measured for each trail. These energy consumption values were transformed to specific energy consumption (SEC) due to the final moisture contents were similar but same. SEC explains the energy requirement to evaporate one gram of water was expressed in watt-hours per gram (Wh/g) and calculated with Eq.9 [43,44]:

$$E_s = \frac{E_c}{W_r} \tag{9}$$

Es: Specific energy consumption (Wh/g)

Ec: Total energy consumption (Wh)

Wr: Evaporated water from madder (g)

Determining of total dye content

The rhizomes dried by microwave drying method were grinded with the help of a blender. 50 mL of distilled water was added to 0.05 g of dry rhizome sample and left to ferment for 12 hours and then kept in a water bath at 70 °C for 1 hour. After this, 50 mL of ethanol was added to the solution, cooled on a shaker and filtered through a 0.2 mm filter. Each filtered sample was made up to 100 mL with 50% ethanol. For standard preparation, 1 mg alizarin and purpurin was dissolved with 100 mL of pure ethanol in a beaker. The standard concentration was equal to 2. The absorbance values of all ethanol extracts and the standard solution were determined with a spectrophotometer (Perkin Elmer UV/VIS Lambda 20) at 450 nm and the total dye content standards based on alizarin and purpurin were calculated using the following formula [40],

$$\text{Total dye content (\%)} = (100/50) * \frac{\text{Standard concentration x Sample absorbance value}}{\text{Standard absorbance value}} \tag{10}$$

Statistical Analysis

Determination of the statistical parameters Minitab and SPSS were used. For estimation of model parameters nonlinear regression analysis performed using Minitab. Statistical multiple comparison test was performed using SPSS. Comparing the model equations R2, χ2, EF and RMSE parameters were evaluated.

Results & Discussion

Determining of drying curves

The change of moisture presence along with time of dry madder samples were dried with microwave. Dry madder was dried from the beginning moisture content of 80.84% present to average 10.86%. The longest drying time occurred at 180 W power density, trails took 41, 18, 10, 9, 8 minutes by increasing microwave power density respectively.

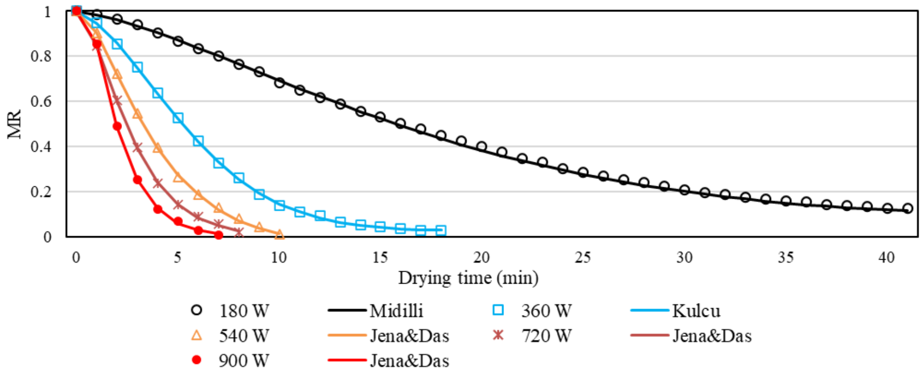

Fig. 1. *Changing of moisture ratio depending microwave power densities*

Increase in power densities reduced drying time as expected (Fig 1.). Dyring condition at 180W it is observed that moisture content isn't get lower, this is probably caused by lack of power. Power density wasn't enough for more drying. Range between the lowest and the highest drying time was 33 minutes. Despite of time reduction indicates important factor, only this factor isn't enough for selecting the method. Also, it needs to be considered that time reduction isn't linear for all conditions, in addition time reduction reduces with increasing power densities.

Drying rate

Drying rate (DR) is the rate at which water is removed from a material. The effectiveness of the drying method can be determined by this parameter. The physical or chemical properties of the material will affect the drying rate. Intervals of each minute of drying rate are calculated and drying curves are drawn. Drying rate curves shows us the smoothness and robustness of drying methods. During trials the highest DR obtained at 900 W with 14.2 g water/g dry matter.min. For better understanding the effect of drying power densities, differences of average drying rates for 180, 360, 540, 720 and 900 W, obtained 0.82, 2.22, 3.90, 4.77, 5.51 g water/g dry matter.min respectively.

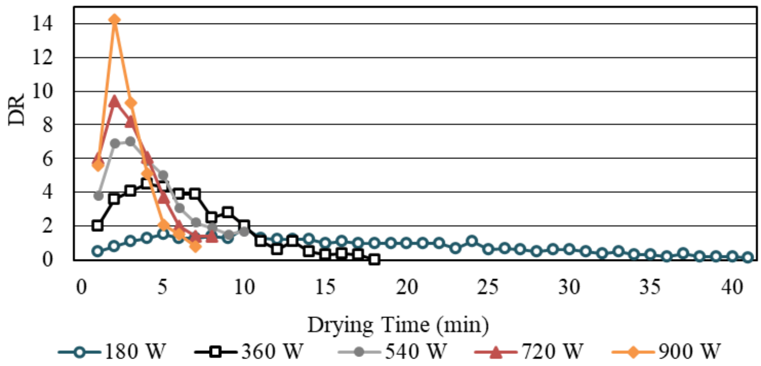

Fig. 2. *Changing of DR with different microwave power densities*

As it seen in Figure 2, the drying rate isn't the highest in the first few minutes. This stage can be called the "warming stage" because the material loses more water mass when it's warmer. After this warming stage the material starts to lose mass rapidly depending on the internal properties. As the moisture content gets lower, the power requirement increases, at some point this requirement and the power density meet, and the drying process ends.

Mathematical Modelling

Mathematical models are applied based on the moisture content obtained during the trials. Thirteen drying models were investigated, selected from previous research. The models' robustness indicators, including R2, χ2, RMSE, and EF parameters, were obtained, and the most appropriate models were selected. The comparison of models is shown in Table 2, and the model constants are given in Table 3. Among the power densities, the Midilli, Kulcu, and Jena&Das models showed the best fits.

Table 2. Comparison of models at different microwave power densities

Model	**180 W**				**360 W**				**540 W**			
	RMSE	**χ^2**	**R^2**	**EF**	**RMSE**	**χ^2**	**R^2**	**EF**	**RMSE**	**χ^2**	**R^2**	**EF**
1	0.0475	0.0023	0.9945	0.9707	0.2292	0.0591	0.9608	0.42728	0.0721	0.0065	0.9977	0.9413
2	0.0121	0.0001	0.9981	0.9980	0.0103	0.0001	0.9990	0.9988	0.0104	0.0001	0.9987	0.9987
3	0.0110	0.0001	0.9984	0.9984	0.0099	0.0001	0.9990	0.9989	0.0098	0.0001	0.9989	0.9989
4	0.0191	0.0003	0.9952	0.9952	0.0286	0.0009	0.9909	0.9910	0.0129	0.0002	0.9980	0.9981
5	0.0182	0.0003	0.9956	0.9956	0.0077	$7.6E^{-05}$	0.9993	0.9993	0.0112	0.0002	0.9986	0.9985
6	0.0298	0.0009	0.9917	0.9884	0.0486	0.0028	0.9813	0.9742	0.0383	0.0021	0.9879	0.9834
7	0.0070	$5.4E^{-05}$	0.9993	0.9993	0.0046	$2.7E^{-05}$	0.9997	0.9997	0.0075	$9.4E^{-05}$	0.9993	0.9993
8	0.0261	0.0007	0.9915	0.9911	0.0372	0.0017	0.9863	0.9849	0.0322	0.0017	0.9897	0.9883
9	0.0092	$9.2E^{-05}$	0.9988	0.9988	0.0485	0.0028	0.9817	0.9742	0.0118	0.0001	0.9985	0.9984
10	0.0097	0.0001	0.9987	0.9987	0.0086	$9.6E^{-05}$	0.9991	0.9991	0.0067	$7.5E^{-05}$	0.9994	0.9994
11	0.0191	0.0004	0.9952	0.9952	0.0286	0.0010	0.9909	0.9910	0.0129	0.0002	0.9980	0.9981
12	0.0082	$7.9E^{-05}$	0.9991	0.9991	0.0044	$2.7E^{-05}$	0.9997	0.9997	0.0089	0.0002	0.9990	0.9991

Table 2. Comparison of models at different microwave power densities (continue)

Model	**720 W**				**900 W**			
	RMSE	**χ^2**	**R^2**	**EF**	**RMSE**	**χ^2**	**R^2**	**EF**
1	0.0652	0.0056	0.9970	0.9482	0.0886	0.0110	0.9874	0.9139
2	0.0090	0.0001	0.9990	0.9990	0.0221	0.0006	0.9949	0.9946
3	0.0059	$5.6E^{-05}$	0.9995	0.9995	0.0043	$3.3E^{-05}$	0.9997	0.9997
4	0.0128	0.0002	0.9978	0.9979	0.0085	0.0001	0.9991	0.9991
5	0.0116	0.0002	0.9982	0.9983	0.0073	0.0001	0.9993	0.999
6	0.0412	0.0027	0.9836	0.9792	0.0671	0.0078	0.9600	0.9506
7	0.0057	$6.5E^{-05}$	0.9995	0.9996	0.0042	$4.2E^{-05}$	0.9997	0.9998
8	0.0209	0.0008	0.9950	0.9946	0.0125	0.0003	0.9984	0.9982
9	0.0412	0.0027	0.9840	0.9792	0.0671	0.0078	0.9604	0.9506
10	0.0047	$4.5E^{-05}$	0.9996	0.9997	0.0037	$3.2E^{-05}$	0.9998	0.9998
11	0.0128	0.0003	0.9978	0.9979	0.0085	0.0001	0.9991	0.9991
12	0.0057	0.0001	0.9995	0.9995	0.0046	0.0001	0.9997	0.9997

Table 3. Model coefficients of chosen model

MODEL COEFFİCİENTS												
180 W[M]	a:	0.9972	k:	0.0143	n:	1.4281	b:	0.0014				
360 W[K]	a:	5688	b:	-0.0035	k:	0.0348	n:	1.7319	c:	1.0212	d:	0.0268
540 W[J]	a:	0.8584	k:	0.4339	b:	0.5376	c:	-0.0483				
720 W[J]	a:	0.6715	k:	0.7389	b:	0.9703	c:	$6.4E^{-05}$				
900 W[J]	a:	0.8325	k:	0.9724	b:	0.9992	c:	0.0014				

Upper letters; M, A, and J represents Midilli, Kulcu and Jena&Das respectively.

Color

Color parameters of trails are given in the Table 4. The L*, a*, and b* values of Owen drying of dry madder were determined as 37.14, 15.49, and 20.33, respectively. The L*, a*, b*, C*, and Hue values of dry madder samples were determined in the ranges of 31.99-37.40, 15.49-21.05, 20.33-26.75, 25.56-34.04, and 50.39-54.29 respectively. Variance analysis results indicated significant ($P<0.05$) impact of microwave power density on all color values. Considering the results, general trend occurs except 720 W in L* which brightness increasing the power density causes darkening. This trend occurred because of power density augmentation. Similar findings to the studies on most studies increasing of power density of microwave causes the darkening the product [13, 37, 45]. For dry madder redness is important factor because of its natural color. Therefore "a" values need to be investigated. Increasing of microwave power density as general redness showed reduction and b value which indicates yellowness increased comparing Owen but within microwave trails didn't show great change. Chroma shows the clarity or purity of color, obtained as the highest value of 34.04 at 180W so it was similar with different studies shows by time the homogeneity increases [26]. Dried with Owen has lowest C* value so it can be considered as microwave dries more homogenize comparing to Owen. Powdering process increases the homogeneity and product becomes pure. This purity makes an impact on customers choices. Hue refers to the basic characteristic of a color. While hue specifies the position of colors within the spectrum, other color properties such as brightness and saturation remain still. Increase of hue shows the changing the base color. It can be determined to the specific color by considering 0° to 360° color space.

Energy consumption

Table 4. Comparing the color parameters of drying methods

Drying Method	L*	a*	b*	C*	Hue
Owen	37.14^{a} (±0.37)	15.49^{d} (±0.05)	20.33^{c} (±0.16)	25.56^{c} (±0.15)	52.68^{ab} (±0.17)
180 W	37.40^{a} (±0.53)	21.05^{a} (±0.26)	26.75^{a} (±0.42)	34.04^{a} (±0.49)	51.80^{bc} (±0.09)
360 W	34.61^{ab} (±0.36)	19.99^{ab} (±0.65)	24.33^{ab} (±0.46)	31.49^{ab} (±0.76)	50.60^{c} (±0.45)
540 W	33.45^{ab} (±2.77)	19.85^{ab} (±0.03)	24.04^{ab} (±1.05)	31.18^{ab} (±0.83)	50.41^{c} (±1.17)
720 W	37.65^{a} (±0.47)	17.45^{c} (±0.52)	24.28^{ab} (±0.78)	29.90^{b} (±0.94)	54.29^{a} (±0.09)
900 W	31.99^{b} (±0.73)	18.87^{bc} (±0.24)	22.81^{bc} (±0.40)	29.61^{b} (±0.43)	50.39^{c} (±0.36)

Energy consumptions transformed to the specific energy consumptions for better comparison, due to the final moisture was similar but not exact in any of the trials. Energy consumptions have determined by real time measuring. (Fig 3). Energy requirements for drying are ranged between 3.12 to 4.82 Whg-1 the total difference with the lowest and highest consumption was 54%. Energy consumptions are expected as linear depends on previous research [6, 45]. However, consumptions are not showing linearity, the lowest energy consumption occurs at 540 W power density. Hence the optimum specific energy value should be between 540 W to 720 W. Main influence that effects energy consumption is drying time. Increase the power density had significant impact until 540 W densities on drying time, after that decreasing in drying time has less effect than previous increases.

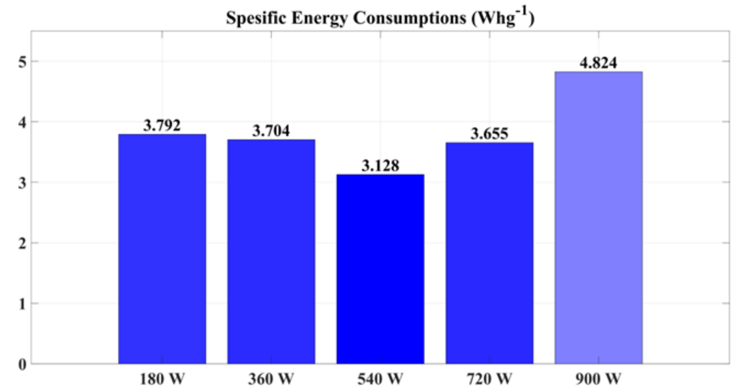

Fig. 3. *Changing of specific energy consumption with different microwave power densities*

Total Dye Content

The variation of total dye content according to different drying methods is given in Fig 4. At different microwave drying power densities, the dye content varied between 1.75- 2.32%. The highest dye content was obtained at drying with 180 W. Baydar and Karadogan [7] used three different propagation methods (seed, seedling and rhizome cuttings) and 5 different propagation methods (direct rhizome planting in autumn, direct rhizome planting in spring, seed sowing in autumn, seed sowing in spring and seedling planting in spring).

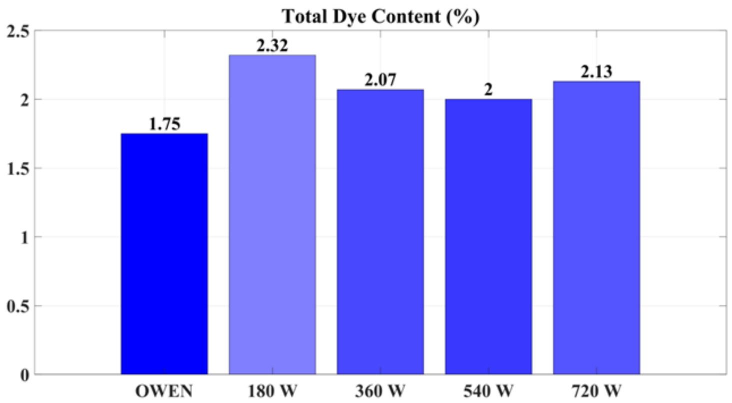

Fig. 4. *Changing of total dye content with different microwave power densities*

Conclusions

The effects of some parameters were examined on madder by drying with microwave. One of the most important parameter on this study was the total dye content because it directly affects yield. Considering the total dye content along the microwave drying with 180 W is recommended. On the other hand, energy consumption is an important factor for selecting the drying power level. These two topic needs to be investigated because these are the main reason for manufactural preferences applications. Energy consumptions are crucial factor for sustainability the production and profitability. Possibilities of reduction in costs are popular subject nowadays. Reducing the energy consumptions performs to increase the yield. Even though energy consumption is the lowest at 540 W power level the total dye content is more important for this study. Additionally, color didn't variate too much in terms of color parameters. Generally dried material darkens with increasing power density as a result of unwanted change occurs. Therefore, drying at lower power densities supports this statement. The total dye content and energy consumptions are foremost subject. The variation of energy consumption between 180 W (3.792 Whg-1) and 540 W (3.128 Whg-1) are 21% nevertheless variation of total dye content between 180 W (2.32%) and 540W (2%) was 16%. It is obvious that the main purpose of this study was produce the most total dye content. Therefore, it is recommended drying should be done at 180W. Further studies the effects of different type of drying methods should be investigated in order to obtain higher total dye content. Pre-treatments also can affect the change of either energy consumptions or total dye content as well.

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