EXTRACTION KINETICS OF MULBERRY (*Morus alba*)

KURU DUT (*Morus alba*) Ekstraksiyonunun Kinetiği

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ABSTRACT: In this study, effects of temperature and time on soluble solids extraction in dried mulberry were determined. The relation between extraction and temperature was described with Arrhenius equation. Activation energy values, which depend on extraction replicates, have showed changes between 164.232-0.787 kcal/g.mol. In addition, values of $1/T.10^3$ at 293°K, 323°K and 358°K were found as 2.79, 3.09 and 3.41, respectively.

ÖZET: Bu çalışmada, kuru dutun çözünür kurumadde ekstraksiyonu üzerine sıcaklık ve sürenin etkisi belirlenmiştir. Ekstraksiyonun sıcaklıkla ilişkisi Arrhenius bağıntısıyla tanımlanmıştır. Aktivasyon enerjisi değerleri ekstraksiyon tekrarına bağlı olarak 164, 232-0.787 kcal/g.mol K arasında değişim göstermiştir. Ayrıca $1/T.10^3$ değerleri, 293 , 323 ve 358 K sıcaklık derecelerinde sırasıyla 2.79, 3.09 ve 3.41 olarak saptanmıştır.

INTRODUCTION

Mulberry, widely grown in Asia, has been consumed not only as a fresh or dried form but also as syrup, pekmez (traditional fruit juice concentrate) and pestil (EKŞİ and ARTIK 1984).

Expression is the main application to obtain fruit juice from fruits. In case of expressing of high soluble solids containing fruits, a considerable amount of soluble content remains in pomace and therefore yield diminishes significantly.

In order to increasing yield, pressing followed by an extraction process is mostly preferred. But in some cases only extraction process is applied, so, insoluble solids are not passed to the extract and fruit juice can be clarified more succesfully (FELLOWS, 1988; CHEN, 1993).

Industrial drying process of mulberry involves the steps of handling, washing, soaking in sulphite solution (0.5 % potassium metabisulphite for 5 minutes) and drying in a suitable equipment (AKSU and NAS, 1996).

In northern regions of Pakistan, mulberries are the second abundant fruits, after apricots. Mulberries are found as black and white varieties. Black mulberries can be sun dried for consumption in winter, but the white varieties is not suitable to sun drying and is mainly eaten fresh; more than 60 % of the white mulberry crop is wasted due to lack of knowledge of preservation techniques (MUHAMMAD et al., 1996).

In a research, four cultivars of mulberry, Morus sp. CVS Mavromoumia (red-frutied), Mavri (black-fruited), Rodini (purple-fruited) and Aspri (white-fruited) were harvested at three stages of harvest maturity (immature, mature and fully mature). The fruit were evaluated for fresh weight, firmness, total soluble solids, titratable acidity and anthocyanin content and composition. Mavromournia mulberries significantly differed when they are compared to the other cultivars for all quality characteristics and especially in titratable acidity and anthocyanin content Cyanidin-3-glucorutinoside is responsible for about 60 % of the fruit coloration (GERASOPOULOS, 1997).

In traditional pekmez production, 1 part of water is mixed with 2-3 parts of fresh mulberry and boiled aproximately 60 minutes. After pressing, raw juice is evaporated up to 75 brix under atmospheric pressure. In this process, considerable amount of soluble solids remains in pomace, and due to the effects of long heating period, quality diminishes significantly. On the contrary of traditional method, mulberry juice obtained by using extraction method contains more flavor compounds and is more desirable (AKSU and NAS, 1996).

Pekmez, one of the traditional foods in Turkey, is produced by using grape, mulberry, apple and fig. Although pekmez is usually extracted from grape (ARTIK, 1977a) mulberry is used, as well. But, mulberry pekmez is preferred to the others due to its high aromatic content and low level of acidity, also no need to deacidification process. Chemical compositions of 20 of mulberry pekmez produced in Turkey is shown in Table 1 (AKSU and NAS, 1996). Pekmez production from fresh mulberry (traditional method) and dried mulberry (industrial method) were outlined in Figure 1a and Figure 1b, respectively.

Table 1. Chemical Composition of Mulberry Pekmez (n = 20) (AKSU and NAS, 1996)

CONTENT	VARIATION			STANDARD DEVIATION	VARIATION COEFFICIENT (%)
	Min	Max	Mean		
Total solids (%)	61.10	76.00	70.19	3.24	1.03
Water (%)	16.80	36.90	29.22	4.10	3.14
Total sugar (%)	7.89	70.56	57.20	17.55	6.85
Reducing sugar (%)	35.07	61.48	51.80	7.66	3.30
Sucrose (%)	2.78	20.79	10.49	4.87	10.39
pH	5.35	6.64	5.77	0.27	1.05
Titratable acidity (%)	0.18	0.71	0.44	0.12	6.04
Ascorbic acid (mg/100 g)	12.00	26.00	17.95	4.20	5.23
Total ash (%)	0.26	1.28	0.88	0.33	8.42
Protein (%)	1.50	2.05	1.71	0.16	2.03

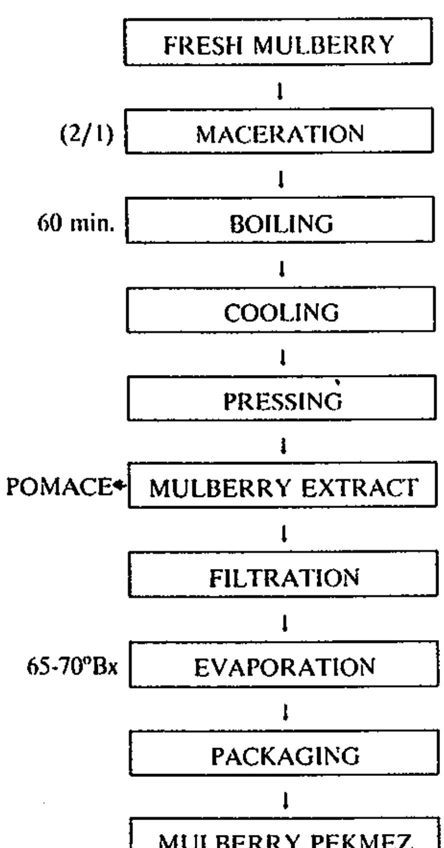
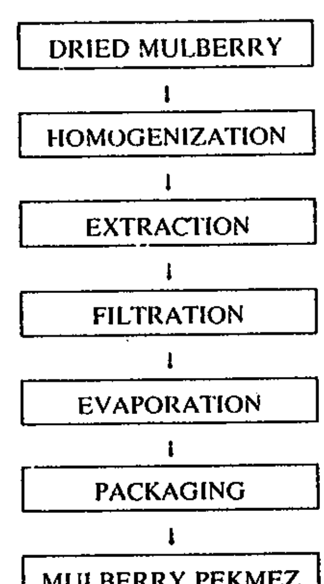


Figure 1a. Mulberry pekmez production outline (Traditional method)



Figüre 1b. Mulberry pekmez production outline (Industrial method)

MATERIAL

Four different dried commercial mulberry samples were obtained from local markets in Antalya. Samples were homogenized in a mortar and used as material. Homogenized samples are used directly for determination of total soluble solids and total ash content. The sample diluted with distilled water (1/10 ratio) and used for the determination of titratable acidity and of pH.

METHODS

Chemical Analyses

Titratable acidity, total ash content and soluble solids content were determined according to IFJU (International Federation of Fruit Juice Producers) methods No 3, No 9 and No 8, respectively (BIELIG et al., 1987). pH was determined potentiometrically and individual sugars (glucose, fructose and saccharose) were determined by using Boehringer test kits (ANONYMOUS, 1989).

Extraction of Mulberry

Temperature and time were studied as parameters on mulberry extraction (GENTCHEV and KLIJANOV, 1988; BAYKUL, 1993; KARKACIER and ARTIK, 1995). 10 g of dried mulberry and 90 ml of water were mixed and extracted in 5 stages and 5 repetitions. Balanced soluble solid point and amount of extract and pomace weight were defined at the end of each application.

Determination of Extraction Conditions at Different Temperatures

Mulberry extraction were performed at 20°C, 50°C and 85°C (KARKACIER and ARTIK, 1995). At these temperatures, extractions were applied as 5 stages and 5 repetitions, and balanced soluble solids were than measured.

Kinetic Parameters

Kinetic parameters such as reaction rate constant, activation energy and frequency factor were calculated according to LABUZA and RIBOH (1982).

RESULTS AND DISCUSSION

Some chemical properties of dried mulberry samples are given in Table 2. Average of total solids and sugar content were found as 89.2 % and 84.5 %, respectively. Sucrose was found as main sugar in mulberry, likewise other berries. Fructose-glucose ratio was around 1 in all samples. pH values were rather high because of the low level of acidity.

Relationship Between Extraction Time and Soluble Solids

During the extraction which continued about 120 minutes, soluble solids were measured on 5 minute intervals. Relationship between extraction time and soluble solids were established by Least Square Method (LABUZA and RIBOH, 1982) and the equations were obtained by using "a" and "K" values as followed;

$$y = 0.9477 + 0.0466x, \quad r^2 = 0.9615$$

Table 2. Some Chemical Properties Of Dried Mullberry Samples (n = 4)

CONTENT	VARIATION			STANDARD DEVIATION	VARIATION COEFFICIENT (%)
	Min	Max	Mean		
Total solids (%)	88.1	91.0	89.2	1.293	1.44
Water (%)	9.0	12.5	11.1	1.535	13.83
Titratable acidity (%)	0.03	0.04	0.04	0.006	18.10
pH value	6.6	6.8	6.7	0.099	14.90
Total sugar (%)	82.4	86.9	84.5	1.886	2.23
Glucose (%)	22.0	24.9	23.6	1.213	5.13
Fructose (%)	21.9	23.4	22.5	0.681	3.02
Sucrose (%)	34.1	40.9	38.4	3.040	7.92
Glucose / Fructose	1.1	1.1	1.1	0.003	0.27
Sucrose/Glucose	1.4	1.9	1.6	0.199	12.22
Total ash (%)	2.5	3.0	1.8	0.224	8.20

Variation of Soluble Solids at Different Temperatures

Determined soluble solids at every stages are given in Table 3, and the relationship between extraction repetition and soluble solids shown in Figure 2, Figure 3 and Figure 4.

Table 3. Soluble Solids Content On Various Stages Of Repetition And Temperatures

STAGE	TEMPERATURE														
	20°C					50°C					85°C				
	Repetition					Repetition					Repetition				
	1	2	3	4	5	1	2	3	4	5	1	2	3	4	5
1	1.4	1.0	0.5	0.4	0.2	2.7	1.7	0.7	0.4	0.1	4.4	2.5	1.3	0.6	0.2
2	3.0	1.8	1.4	0.9	0.4	6.3	3.7	2.6	1.6	1.2	9.6	5.7	3.2	1.6	0.9
3	4.3	2.9	2.1	1.3	0.7	9.7	6.5	4.5	3.5	1.9	16.3	9.2	5.4	3.3	1.5
4	4.8	3.9	2.7	1.5	1.5	14.4	8.9	6.7	4.6	3.1	22.7	13.2	8.5	4.4	2.3
5	6.1	5.0	3.8	1.8	2.0	18.0	12.0	8.0	6.0	4.0	30.9	17.9	10.6	5.9	3.3

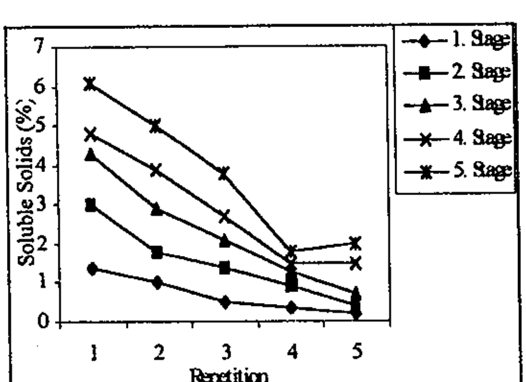


Fig. 2. Relationship between extraction repetition and soluble solids (20°C)

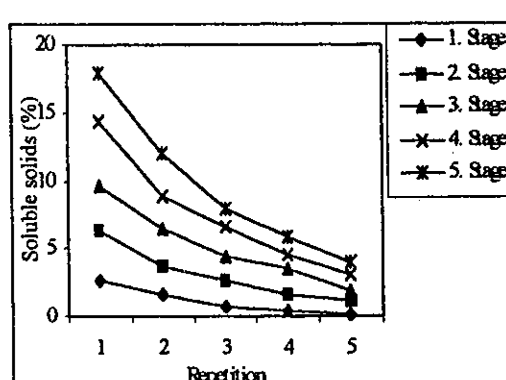


Fig. 3. Relationship between extraction repetition and soluble solids (50°C)

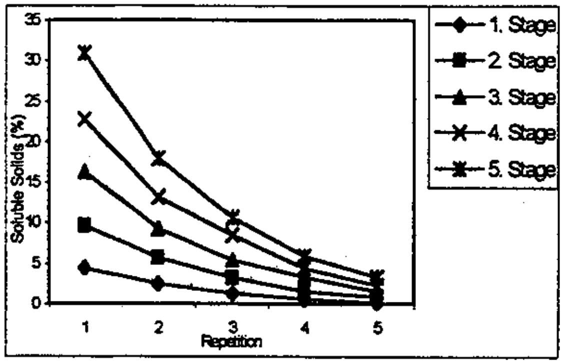


Fig. 4 : Relationship between extraction repetition and soluble solids (85°C)

Table 4 shows the kinetic parameters of extraction, extraction line equation of every repetition reaction rate constant and correlation coefficient.

Table 4 : Kinetic Parameters at Different Temperatures And Repetition Stages

TEMPERATURE	REPETITION	EQUATION	r^2
20°C	1	y = 0.528 + 1.128x	0.989
	2	y = –0.270 + 1.008x	0.959
	3	y = –0.256 + 0.788x	0.965
	4	y = –0.292 + 0.576x	0.930
	5	y = –0.449 + 0.471x	0.910
50°C	1	y = –1.390 + 3.87x	0.964
	2	y = –1.180 + 2.58x	0.957
	3	y = –1.15 + 1.88x	0.978
	4	y = –1.04 + 1.42x	0.973
	5	y = –0.85 + 0.97x	0.970
85°C	1	y = –3.05 + 6.61x	0.954
	2	y = –1.35 + 3.71x	0.953
	3	y = –1.44 + 2.405x	0956
	4	y = –0.91 + 1.34x	0.963
	5	y = –0.64 + 0.76x	0.951

A positive correlation observed between increased temperature and K values (Figure 5). Increased numbers of repetition on extraction resulted decreased soluble solids content and therefore K values were also decreased correlation coefficient was quite high on this relationship.

At different temperatures calculated activation energy, $1/T.10^3$ and L_n K values were used to perform Arrhenius Plot (Figure 6).

Activation energy values of extraction at different temperatures are shown in Table 5. These activa-

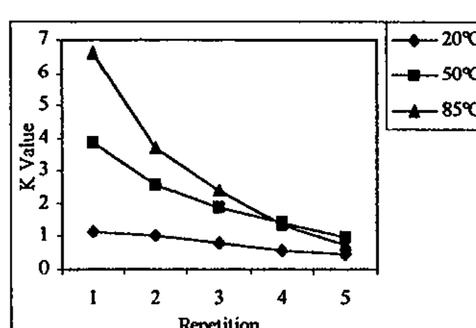


Fig. 5. Relationship between K value and repetitions

tion energy values explains the relationship between requirement of energy and soluble solid units. As a result of this relationship, activation energy requirement decreased when extraction repetition were increased. This was due to decreasing amount of soluble solids and increased amounts of solvent.

It is a well known fact that increased amounts of solvent results increased soluble solids. But, as a result of this application, activation energy diminishes, and evaporation of solvent becomes a problem.

Activation energy value may be used for finding out the economically optimum point. Also, soluble solids remained in pomace, cost of energy and pressing time must be considered.

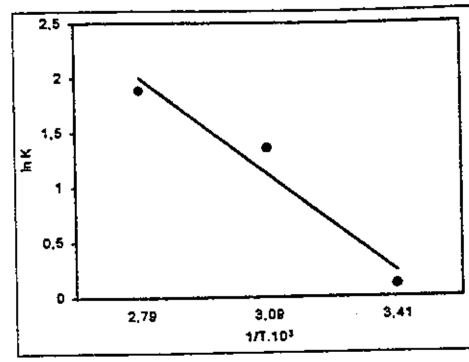


Fig. 6. Arrhenius Plot for mulberry extraction

Table 5 : Activation Energy Values at Various Temperatures

REPETITION	EQUATIONS	a VALUE	K VALUE	ACTIVATION ENERGY Kcal/g mol°K
1	y = 254.96 + 82.65x103x	254.96	82.65x103	164.232
2	y = 7.288-2.111x10-3x	7.288	-2.111x103	4.1924
3	y = 3.7848-1.356x103x	3.7848	-1.356x103	2.6934
4	y = -0.8999+0.4193x103x	-0.8999	0.4193x103	0.8327
5	y = -1.2032+0.787x103x	-1.2032	0.787x103	0.787

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