



Liquorice (*Glycyrrhiza glabra*): Production of instant soluble microcapsules

Meyan (*Glycyrrhiza glabra*): Çözünebilir hazır toz üretimi

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ABSTRACT

In this study, bioactive compounds were extracted from liquorice (*Glycyrrhiza glabra*) and converted to instant form by the spray dryer. The four different feed emulsions were prepared with liquorice extract as core material, 12% maltodextrin (DE 18-20)/gum Arabic (MD/GA) combination as the coating material. The ratio of core to wall was 1:3. The MD and GA ratio significantly ($p<0.05$) affected on some parameters of final products. The encapsulation yield and efficiency were observed to vary between range of 46.30-64.11% and 98.15-98.62%, respectively. The encapsulation yield of the feed emulsion of FE₄ (MD/GA: 2.25/0.75) was the highest in comparison to the values for other feed emulsions. However, there were not find any significant differences between encapsulation entrapment of the feed emulsions ($p>0.05$). The highest total phenolic substances (7.02 mg GAE per g of sample) and antioxidant activity (60.15 mg TEAC per g of sample) were found in FE₁ (MD/GA: 0.75/2.25). This study proves the success of instant liquorice production using a spray drying technique.

Key Words: *Glycyrrhiza glabra*, Powder, Maltodextrin, Gum Arabic, Spray drying

ÖZ

Bu çalışmada meyan kökünden (*Glycyrrhiza glabra*) biyoaktif bileşikler ekstrakte edilmiş ve elde edilen ekstraktlar püskürtmeli kurutma tekniği ile toz forma dönüştürülmüştür. Kurutma yardım materyali olarak maltodekstrin (MD) ve gam arabikin (GA) farklı konsantrasyonları kullanılmıştır. Çekirdek materyalinin kaplama materyaline oranı 1:3 olarak ayarlanmış ve tüm denemelerde sabit tutulmuştur. Kullanılan duvar materyali konsantrasyonu elde edilen son ürünlerin bazı özellikleri üzerine istatistiksel olarak etki olduğu saptanmıştır ($p<0.05$). Enkapsülasyon verimi ve enkapsülasyon etkinliği sırasıyla 46.30-64.11% ve 98.15-98.62% arasında bulunmuştur. FE₄ (MD/GA: 2.25/0.75) emülsiyonunun kapsüllüme verimi diğer emülsiyonlar ile karşılaştırıldığında en yüksek bulunmuştur. Fakat farklı emülsiyonların enkapsülasyon etkinliği üzerine önemli derecede etkileri görülmemiştir ($p>0.05$). En yüksek toplam fenolik madde içeriği (7.02 mg GAE/g toz) ve antioksidan aktivite (60.15 mg TEAC/g toz) FE₁ (MD/GA: 0.75/2.25) emülsiyonunda bulunmuştur. Bu çalışma, püskürtmeli kurutma tekniği kullanarak hazır meyan kökü üretiminin başarılı bir şekilde gerçekleştirilebileceğini göstermiştir.

Anahtar Kelimeler: *Glycyrrhiza glabra*, Toz, Maltodekstrin, Gam Arabik, Püskürtmeli kurutma

Introduction

Liquorice '*Glycyrrhiza glabra*' belonging to *Fabaceae* is grown in tropical and subtropical regions (Vibha et al., 2009). There are more than

30 *Glycyrrhiza glabra* varieties in the region from the Mediterranean to the east of Europe. The used parts of the plant are roots (*Liquiritae radix*), and extracts (*Liquiritiae succus*) obtained from roots. Liquorice has been used as tea in the non-

alcoholic beverage industry throughout the world. Its extract contains about 500 different useful compounds such as antioxidants, saponin, isoflavone. Therefore, it is consumed widely and also used in the treatment of diseases. Liquorice is evaluated as an enrichment material in different food products and a considerable ingredient of pharmaceutical and cosmetics (Hayoğlu et al., 2017; Tung et al., 2016; Wang et al., 2015).

Liquorice tea is produced by traditional methods and consumed daily. It is served in tea bags containing its root or prepared traditionally and consumed. However, its shelf life is too short. Additionally, it has also a problem that is unpractical like instant coffee. The instant products produced by proper techniques do not exert the aforementioned shortcomings. Spray drying is generally used to produce instant products. It is cheaper than freeze-drying (Moreau and Rosenberg, 1996). Other advantages are low cost, easy handling, preservation of volatile and aroma substances stability (Reineccius, 2004). Previous data regarding the microencapsulation of sour cherry juice (Garofulić et al., 2017) was reported. Similarly, liquorice root extract can be converted to instant form by spray drying technique. Maximum microencapsulation yield (MY), microencapsulation efficiency (ME), and flowing property were determined in emulsion prepared by using 100% maltodextrin as a wall material in our previous study conducted for finding the optimum ratio of gum Arabic to maltodextrin (Başyigit and Hayoğlu, 2019). All properties of powders produced at the optimum point were investigated in this previous study. Additionally, the effect of gum Arabic and maltodextrin on all properties of liquorice powder is essential to discuss in detail. Therefore, we used a binary mix (except %100 maltodextrin) of maltodextrin (DE 18-20) (MD) and gum Arabic (GA) as encapsulating agent for the production of instant liquorice in this study and investigated the changes in Hausner ratio, colour values, water activity, solubility, moisture content, wettability, surface, and total phenolic content and antioxidant activity of final products.

Material and Methods

Materials and chemicals

Liquorice was obtained from a local grower in Şanlıurfa province of Turkey. After drying at ambient conditions for 7 days, it was used for further analysis. The coating materials (MD and GA) were obtained from Smart Chemistry (Izmir, Turkey). The remaining chemicals used in the analyses were purchased from Sigma and Merck (Darmstadt, Germany).

Extraction conditions for phenolics

A 100 ml glass beaker containing 5 gram liquorice and 50 mL water was incubated at 60 °C for 60 min. After centrifuging at 5000 rpm for 5 min, the extract was collected and stored at -18 °C for using in the further analysis (Başyigit and Hayoğlu, 2019).

0.5 g instant liquorice was mixed with 10 mL ethanol for surface phenolic analysis (Zhang et al., 2007), 10 ml water for total phenolic analysis (Içyer, 2012). These mixtures were incubated at ambient conditions for 4 min in a shaker. After centrifuging at 5000 rpm for 5 min, the extract was collected and stored at -18 °C for use in further analysis.

Microencapsulation of liquorice extract

Feed emulsion solid content was adjusted according to total soluble solid content (TSSC) of extract. The total soluble solids content, which was originally 4 g 100 mL⁻¹, was adjusted to 16 g 100 mL⁻¹ by adding MD and GA (Başyigit and Hayoğlu, 2019). The homogenization process was carried out for 5 min by Ultra-Turrax homogenizer (IKA-T18 Basic, Japan). Emulsions were fed as a 100 ml mixture for spray drying. The effects of the MD/GA ratio on the quality parameters of instant liquorice microcapsules were investigated. The concentrations of wall material are given in Table 1.

Microencapsulation was applied by a laboratory-type spray dryer (Buchi-B290, Flawil, Switzerland) with a division diameter of 16.5 cm and a division length of 60 cm. Conditions: air inlet temperature 140 °C, outlet temperature 93±3 °C, the pump speed 600 l h⁻¹, feeding rate 8 ml min⁻¹.

Table 1. The wall material concentrations

Feed Emulsion	TSSC/MD/GA (g/g/g)
FE ₁	1:0.75:2.25
FE ₂	1:0:3
FE ₃	1:1.5:1.5
FE ₄	1:2.25:0.75

TSSC: Total soluble solid content, MD: Maltodextrin, GA: Gum Arabic

Physiochemical properties of microcapsules

Microencapsulation yield and efficiency

MY (Çam et al., 2014) and ME (Içyer, 2012) were calculated depending on the following equations (Eq.1 and Eq.2):

$$MY (\%) = \frac{\text{the weight of final products (g)}}{\text{the weight of initial substanses (g)}} \times 100 \quad (1)$$

$$ME (\%) = \left(1 - \frac{\text{Phenolics on final products surface}}{\text{Total phenolic of final products}} \right) \times 100 \quad (2)$$

Moisture and water activity

The moisture content was performed gravimetrically. The water activity was determined by using the HygroPalm AW1 water activity meter (Rotronic ag, Germany).

Wettability

The wettability of the products was made in accordance with Turchiuli et al. (2005) by measuring the time to disappear from the water surface using 1 g sample (100 ml, 20°C).

Solubility

The solubility of products was performed gravimetrically (Cano-Chauca et al., 2005). Boiling distilled water (50 mL) was mixed with the instant product (0.5 g). After the mixture was vortexed at ambient conditions, centrifuged at 4000 rpm for 5 min. The supernatant (25 mL) was added to a petri dish and kept at 70 °C for 24 hours. The following equation was used to determine the solubility (Eq.3).

$$\text{Solubility (\%)} = \frac{\{(m_2 - m_1) / m_3 \times S_1\}}{S_2} \times 100 \quad (3)$$

Where, m₁, m₂, m₃, S₁, and S₂ are the initial weight of the petri dish (g), the final weight of the petri dish (g), the weight of the product (g), the initial amount of water (mL), and the final amount of water added to the petri dish (mL), respectively.

Density

For the bulk and tapped density, 3 g instant liquorice was put inside the measuring cylinder (25 mL) and the cylinder was tapped until reached constant volume on a flat surface. The first volume and second volume were used to determine the bulk and tapped density respectively (Tatar et al., 2014). The bulk and tapped density were determined as g/mL by the following equation (Eq.4 and Eq.5):

$$\text{Bulk density } (\rho_B) = \frac{\text{Products mass}}{\text{Volume of products}} \quad (4)$$

$$\text{Tapped density } (\rho_T) = \frac{\text{Products mass}}{\text{Final volume of products}} \quad (5)$$

Hausner Ratio

Hausner ratio (Turchiuli et al., 2005) was calculated by the formula below (Eq.6):

$$\text{Hausner ratio (HR)} = \frac{\rho_T}{\rho_B} \quad (6)$$

Where, ρ_T and ρ_B are the tapped density and the bulk density, respectively.

Surface and total phenolic contents

Surface and total phenolic content of final products were estimated spectrophotometrically using the Folin-Ciocalteu assay in accordance with Singleton et al. (1999).

ABTS radical scavenging activity of instant liquorice

ABTS(2,2'-azino-bis(3-ethyl-benzothiazoline-6-sulfonic acid) radical scavenging activity of products was made according to Dai et al. (2010).

Colour analysis

The colour difference has measured with Colour Quest XE colorimeter (Reston, VA, USA) in accordance with Toğrul and Hayoğlu (2020). The CIE L* (lightness), a* (redness), b* (yellowness), C (chroma), and h° (hue angle) values were determined.

Statistical analysis

Analyses were made in two parallel and three replications. The obtained values were subjected

to variance analysis (ANOVA) using the SPSS 22.0 package program and evaluated at the significance level of $p < 0.05$ according to the Tukey HSD test.

Results and Discussion

Encapsulation yield

The MY is presented in Table 2. The microencapsulation yield of FE₂, FE₁, FE₃, and FE₄ was 42.30, 56.90, 60.96, and 64.11%,

respectively. All feed emulsion was found more than 50% except FE₂. It is critical that the microencapsulation yield for effective drying is greater than 50% (Bhandari et al., 1997). The encapsulation yield determined was different than those by reported (Raisin juice) Papadakis et al. (2006), and (Pomegranate juice) Vardin and Yaşar (2012). These authors indicated that there were positive correlations between encapsulation yield and the amount of wall material.

Table 2. Effects of wall concentration on yield, efficiency, moisture content, Hausner ratio, water activity, solubility and wettability of final product

Feed Emulsion	Yield (%)	Efficiency (%)	Moisture content (%)	Water activity	Hausner ratio	Solubility (%)	Wettability (s)
FE ₁	56.60	98.55±0.36 ^a	2.09±0.01 ^a	0.21±0.01 ^a	1.53±0.01 ^a	99.40±0.01 ^b	212±3.54 ^a
FE ₂	42.30	98.37±0.03 ^a	1.47±0.01 ^d	0.16±0.01 ^b	1.40±0.01 ^d	98.69±0.01 ^d	234±10.61 ^a
FE ₃	60.96	98.30±0.04 ^a	1.74±0.02 ^b	0.20±0.01 ^a	1.50±0.01 ^b	99.22±0.01 ^c	217±3.54 ^a
FE ₄	64.11	98.60±0.48 ^a	1.58±0.01 ^c	0.17±0.01 ^b	1.43±0.01 ^c	99.50±0.01 ^a	231±1.41 ^a

*The results are expressed as a mean ± standard deviation of three replicates. Different lowercase letters (^{a-d}) in the same column represent the differences among the means ($p < 0.05$)

Moisture content and water activity

Statistically significant differences ($p < 0.05$) were observed among the moisture content of feed emulsions (Table 2). As expected, there was also a significant difference ($p < 0.05$) among the water activity of final products. The differences could be attributed to the different emulsion viscosity (Premi and Sharma, 2017). When the MD in the emulsion is high, moisture content decreases (Nhu Quynh et al., 2016). The moisture content ranged between 1.47 and 2.09 % which was sufficiently necessary to make food powder microbiologically safe. The moisture content less than 5% is considered as the criteria for the instant product produced by spray drying. Therefore, the instant liquorice could have a long shelf life as microbiological and chemical degradation are difficult under 0.3 of water activity (Bicudo et al., 2015).

Hausner Ratio

The parameter plays an important role in the packaging and transport of the product. The lower the Hausner ratio the higher the flowability. The FE₂ exhibited the highest flowability, followed by FE₄ > FE₃ > FE₁ (Table 2). All samples showed

different flowability (week or too week) because of van der Waals and electrostatic forces core material (Tze et al., 2012).

Solubility and wettability

Solubility is an indication of the ability to form a solution or suspension in water for spray dried products (Bicudo et al., 2015). The FE₄ exhibited higher solubility compared to FE₁, FE₂, and FE₃. The solubility of products ranged from 98.69 to 99.50%. The results were more than those by reported Sanchez-Reinoso et al. (2017) while working on spray drying of cocoa aroma.

Wettability is known as the water absorption ability of encapsulated powder (Gaiani et al., 2007). The wettability values varied from 212 to 234 sec. The results were in agreement with Fernandes et al. (2013) while working with spray dried rosemary essential oil (155-481 sec).

Antioxidant activity, encapsulation efficiency, surface phenolic content and total phenolic content

Figure 1 shows the antioxidant activity measured by the ABTS method. The antioxidant activities were found to be in following order: FE₁

(60.15 mg TEAC g⁻¹) > FE₂ (56.54 mg TEAC g⁻¹) > FE₄ (51.48 mg TEAC g⁻¹) > FE₃ (50.13 mg TEAC g⁻¹). The same order was also observed for the total phenolic content of products (Figure 2). The total phenolic content of products was found to vary between ranges of 6.45-7.02 mg GAE g⁻¹. Due to the lack of information in the literature about liquorice encapsulation, we have compared the

results with those of other products. Previous studies on total phenolic content and antioxidant activity of different instant products such as pomegranate juice (8.80 mg GAE g⁻¹) (Miravet et al. 2016), *Mentha spicata* (81.12 mg GAE g⁻¹), and *Mentha piperita* (69.85 mg GAE g⁻¹) (Alaşalvar, 2017) and pomegranate juice (6.03 mg TEAC g⁻¹) (Miravet et al., 2016) were reported.

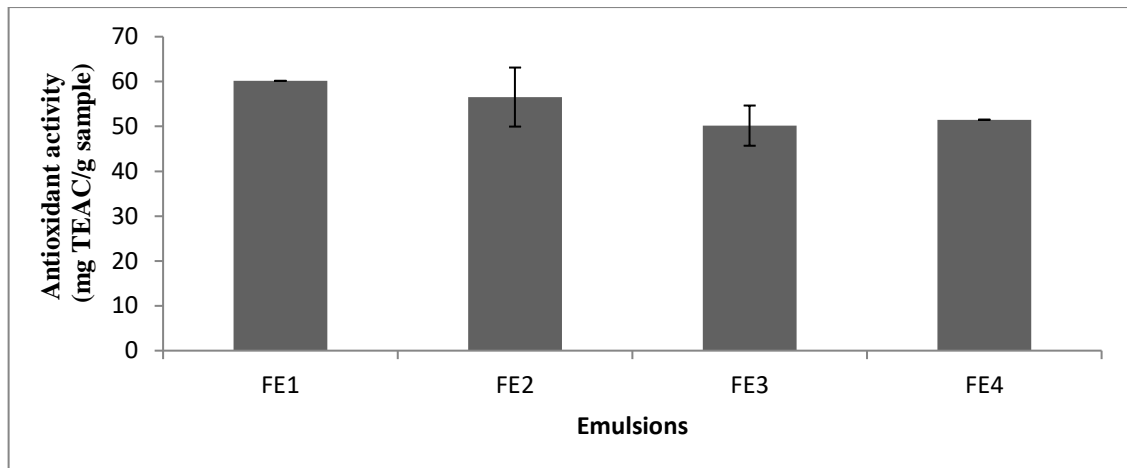


Figure 1. Antioxidant activity of final products

The lower the surface phenolic contents the higher efficiency and the higher stability. Encapsulation efficiency was not significantly ($p>0.05$) influenced with wall material concentration (Table 2). Because, liquorice contains starch and different gums. The

structures behaved like wall material and protected the bioactive compound. These results indicated that the surface phenolic content depends on not only wall material but also core material.

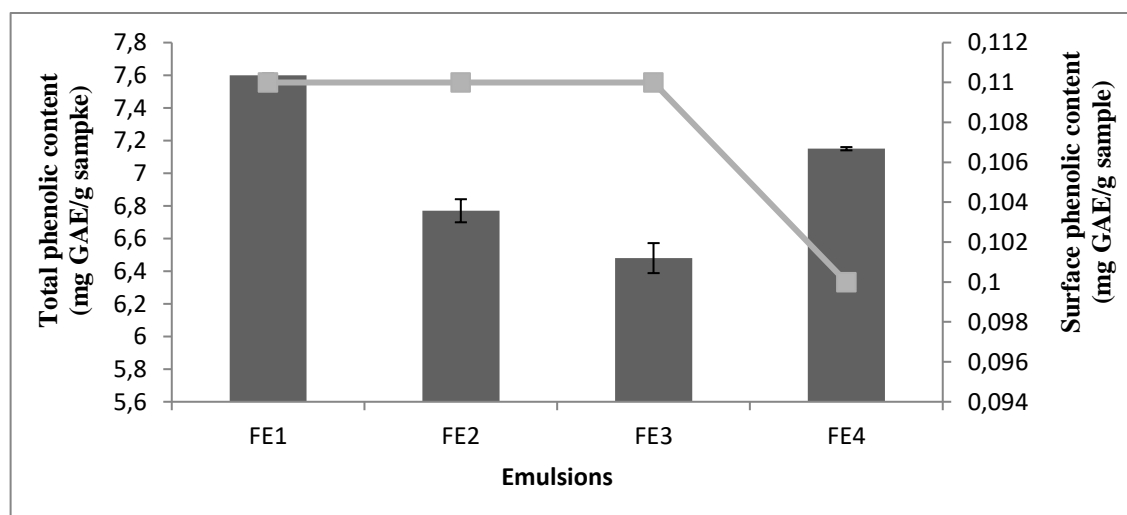


Figure 2. Total phenolic and surface phenolic content of final products
GAE: Gallic acid equivalent, TEAC: Trolox equivalent antioxidant activity

Colour

Lightness values and hue angle (more yellow) of FE₂ were significantly higher ($p<0.05$) than the other products (Table 3). On the other hand, the FE₄ had also lower redness (a^*). The decrease in

MD concentration leads to a reduction in redness, blueness, and Chroma for instant liquorice because of the white color of MD (Kang et al., 2019). The hue angle was higher in FE₂ than the others, indicating that FE₂ was less red compared to the

others. Furthermore, the lowest chroma was found for instant products obtained by formulation FE₂ with GA as wall material. The

results indicated that the wall material concentration and type were significant ($p<0.05$) on product colour.

Table 3. Effects of wall concentration on final product colour

Feed Emulsion	L*	a*	b*	Chroma	Hue angle
FE ₁	74.83±0.01 ^b	3.11±0.01 ^c	26.38±0.01 ^{bc}	26.75±0.27 ^{bc}	83.32±0.06 ^b
FE ₂	75.87±0.01 ^a	2.55±0.49 ^d	25.15±0.75 ^c	25.27±0.75 ^c	84.22±0.06 ^a
FE ₃	74.58±0.14 ^b	3.26±0.01 ^b	27.83±0.09 ^{ab}	28.02±0.09 ^{ab}	83.30±0.01 ^b
FE ₄	73.28±1.20 ^c	3.48±0.01 ^a	28.91±0.01 ^a	29.55±0.61 ^a	83.03±0.15 ^b

*The results are expressed as a mean ± standard deviation of three replicates. Different lowercase letters (^{a-d}) in the same column represent the differences among the means ($p<0.05$)

Conclusion

Liquorice extract has short shelf life resulting from a high microbial load and preparing its extract is not practical. However, instant liquorice showed that low moisture content (1.47-2.09 %) and water activity (below 0.3). Therefore, the products were microbiologically safe range. Preparation of instant liquorice such as instant coffee, tea is not taken time. Furthermore, instant liquorice can be produced using MD and GA. On the other hand, it could be produced by using different wall materials except for MD and GA and in the different spray drying conditions in future studies.

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Author Contribution: Bülent BAŞYİĞİT: Formal analysis, Conceptualization, Data curation, Investigation, Methodology, Software, Writing—original draft. İbrahim HAYOĞLU: Conceptualization, Data curation, Investigation, Project administration, Resources, Supervision, Writing – original draft, Writing— review & editing. Gülbahar AKYAR: Formal analysis, Investigation, Methodology. Sara BULUT: Formal analysis, Investigation, Methodology. Ömer Faruk GAMLI: Visualization, Writing – original draft. Ahmet Ferit ATASOY: Software, Writing – original draft.

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