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Research Article/Araştırma Makalesi

The influence of anti-caking agents on powder flow properties of ready-to-drink coffee during storage

Topaklanma önleyici maddelerin içime hazır kahvenin depolama sırasında toz akış özelliklerine etkisi

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ABSTRACT

Anti-caking agents are used in powder product formulations to prevent caking and to positively influence consumer preferences. In this study, ready-to-drink coffees, which are highly consumed were preferred and silicon dioxide (SiO_2), maltodextrin (MD), glycerol monostearate (GMS), tricalcium phosphate (TCP) additives were added to the product formulations as anti-caking agents. Caking, cohesion and powder flow rate dependence (PFSD) tests were carried out on the 1st, 15th, 30th, 30th, 45th, 60th, 75th and 90th days and Scanned Electron Microscope (SEM) images were taken. In this way, the performances of anti-caking agents on instant coffee powder products during storage processes were determined. At the end of the study, it was determined that the powder flow properties of the anti-caking agents during the storage process were different. While the mean cake strength value of the control coffee sample was determined in the range of 93.90-159.63 g mm during the storage period, no caking was observed in the MD sample. The SEM imaging results of the samples are similar to the powder flow measurement values.

Key Words: Anti-caking agent, ready-to-drink coffee, powder flow, caking

ÖZ

Topaklanma önleyici ajanlar toz ürün formülasyonlarında kekleşmeyi önleme ve tüketici tercihlerini olumlu yönde etkileme amaçlarıyla kullanılmaktadır. Bu çalışmada tüketimi oldukça fazla olan içime hazır toz kahve karışımları tercih edilmiş ve ürün formülasyonlarına silikon dioksit (SiO2), maltodekstrin (MD), gliserol monostearat (GMS), trikalsiyum fosfat (TCP) katkı maddeleri topaklanma önleyici ajanlar olarak eklenmiştir. Elde edilen toz karışım kahve çeşitleri ve kontrol grubu kahve örneğinin 1.,15.,30.,45.,60.,75 ve 90. günlerde kekleşme, kohezyon ve toz akış hızı bağımlılık (PFSD) testleri yapılmış ve Taramalı Elektron Mikroskobu (SEM) görüntüleri alınmıştır. Bu sayede topaklanma önleyici ajanların depolama süreçlerinde hazır toz kahve ürününde gösterdikleri performanslar tespit edilmiştir. Çalışma sonunda topaklanma önleyici ajanların depolama sürecinde gösterdiği toz akış özelliklerinin farklı olduğu belirlenmiştir. Kontrol kahve örneğinin depolama süresi boyunca ortalama kek gücü değeri 93.90-159.63 g mm aralığında belirlenirken MD örneğinde hiç kekleşme gözlenmemiştir. Örneklerim SEM görüntüleme sonuçları da toz akış ölçüm değerleri ile benzerlik göstermektedir.

Anahtar Kelimeler: Topaklanma önleyici ajan, hazır kahve, toz akış, kekleşme

Introduction

In these days, many of the foods and ingredients on the market are supplied in powder form to facilitate handling and processing and to increase the stability of the ingredients (Fitzpatrick et al., 2005). The measurement of powder flow properties of foods is of great importance as it gives important clues about the behavior of powder during storage, transportation and processing. Powder flow characteristics are important in handling and processing operations such as flow from hoppers and silos, transport, mixing, compaction and packaging (Fitzpatrick et al., 2004). It has gain importance to know the properties of powders due to the increasing value of products, the complexity of processing unit operations and the formulations of new products (Zou et al., 2002).

The caking or agglomeration of free-flowing powder into lumps causes major problems in the processing of solid hygroscopic particulate materials. Therefore, it has been investigated for a long time to solve this problem (Adams et al., 1929; Bode et al., 2015). Caking in food powders is a common problem that can contribute to reduced product quality, functionality and shelf life (Descamps et al., 2013; Lipasek et al., 2012). It can occur during processing, transport or storage and therefore can be a problem for both production lines and consumers (Lipasek et al., 2012). In the case of caking in powder products, additional processing steps are often needed in which the powder is broken down before adding the raw material to silos or bunkers (Larsson, 2016). From the consumer's point of view, the formation of agglomerates and the reduced flowability of the powder can cause difficulties in emptying the jar of the powder products or the formation of lumps in the powdered food when emptying a bag with the powdered ingredients. Therefore, anti-caking agents are utilized to prevent consumer dissatisfaction and to increase the flowability and processing of powders. Anti-caking agents can function by one of several mechanisms. One of these mechanisms is to compete with host powder for moisture, the other is to create protective barriers against moisture on the surface of hygroscopic particles or physical barriers between particles, another mechanism is to smooth surfaces to eliminate inter-particle friction; and to inhibit crystal growth, which is important in solid bridge formation (Lipasek et al., 2012).

Only a few publications are dealing with the effect of anti-caking agents on food powders (Akins, 2002; Nurhadi et al., 2017; Pui et al., 2020; Sharma et al., 2009), on the other hand, the comparison of anti-caking agents with each other and the effects of anti-caking agents on cohesion values and powder flow speed dependency values have not been addressed. It was hypothesized that the addition of anti-caking agents could prevent caking and improve powder flow properties of ready-to-drink coffees. Accordingly, the aims of the present paper were i) to investigate the influences of various anti-caking agents on the powder flow behavior and storage stability of instant coffees, ii) to classify and compare the effect of anti-caking agents, iii) to determine the best anti-caking agents to prevent caking and enhance flowing.

Materials and Methods

Materials and chemicals

Vegetable oil powder (78% fat), which is used as a material in the production of ready-to-use powdered coffee, is procured by Enka Sut A.S., (Konya, Türkiye) and skimmed milk powder was obtained from Pınar Sut Mamulleri San. Inc. (İzmir, Türkiye). Icing sugar and granulated coffee were purchased from local markets. Silicon dioxide (SiO_2 100x, Tito, Turkey), maltodextrin (MD, Sigma Aldrich, USA), glycerol monostearate (GMS, Alfasol, Turkey) and tricalcium phosphate (TCP, Sigma Aldrich, USA) were used as anti-caking agents.

Methods

Preparation of instant coffee powders

For preparing coffee samples 360 g skim milk powder, 100 g powdered sugar, 17 g vegetable oil powder, 80 g coffee and 4.40 g anticaking agent were weighed and mixed, and stored at room temperature until analysis.

Determination of the powder flow behaviors

The flow properties of instant coffee powders were determined by testing the powder flow rate dependence (PFSD test), cohesion and caking (caking test) properties of the products using the Stable Micro System (TA-XT2 Plus, UK). For the analysis, a glass cylinder with a height of 120 mm, an inner diameter of 50 mm and a diameter of 48 mm was used. In addition, a special powder flow probe (Selective Laser Sintered DMLS Stainless Steel 316) with a height of 10 mm and moving in horizontal and vertical planes was used in the analysis. The degree of caking, PFSD and cohesion coefficient values were calculated with Texture Exponent 32 software.

Scanned electron microscopy (SEM)

Scanned Electron Microscope (SEM) (Tescan Mira3, Czech Republic) was used to identify the morphological structures of the instant coffee powders.

Statistical analysis

The ANOVA multiple comparison approach was used to determine statistical analysis of the study's data using the Minitab (Minitab Ltd., Coventry, England). To evaluate the difference between the means, the Tukey ANOVA test was utilized. The statistically significant difference is denoted by $p<0.05$.

Results and Discussion

Powder flow behaviors of instant coffee samples

Cohesion index values

The cohesion index value provides information about the cohesiveness values of the samples. Cohesion is defined as the tendency of powdered food particles to come together and form larger powder areas (Mercan et al., 2018). Flow models that vary depending on the cohesion index of the samples are evaluated based on the flow property table of Benkovic and Bauman (2009). According to the table, if the cohesion index is 19, the flow is defined as hardened, extremely cohesive, while 16-19, 14-16, 11-14 and 11 are expressed as very cohesive, cohesive, easy flowing and free flowing respectively.

Cohesion index values increased in all ready-to-drink coffees samples during the storage process. The interesting thing is that the control coffee sample, without the addition of anti-caking agent, showed an easy flowing value at the beginning of the storage process, but at the end of the storage it moved to the extremely cohesive category and showed a much higher cohesion index value than the other samples. In this case, it can generally be said that the addition of anti-caking reduces the cohesion index value. In particular, the cohesion index values of the coffee sample with the addition of GMS anticaking agent were determined to be the lowest. On the other hand, although the rates of change during the storage process differed, the initial and final day cohesion index values of TCP and MD samples were found to be the same. During 15 days of storage, SiO_2, GMS showed free flowing behavior, while all other samples were determined to be easy flowing. At the end of 90 days of storage, GMS sample showed cohesive behavior, while SiO_2, MD and TCP samples showed very cohesive behavior (Figure 1).

In powder mixtures, all powder flow properties, especially the cohesion index value, vary with the materials added to the product formulation. In addition, various factors such as hygroscopicity, electrostatic activity, porosity, particle size and shape can also change the cohesion index value (Thomas et al., 2004). On the other hand, the addition of anticaking agent and variety change also effected the cohesion index values.

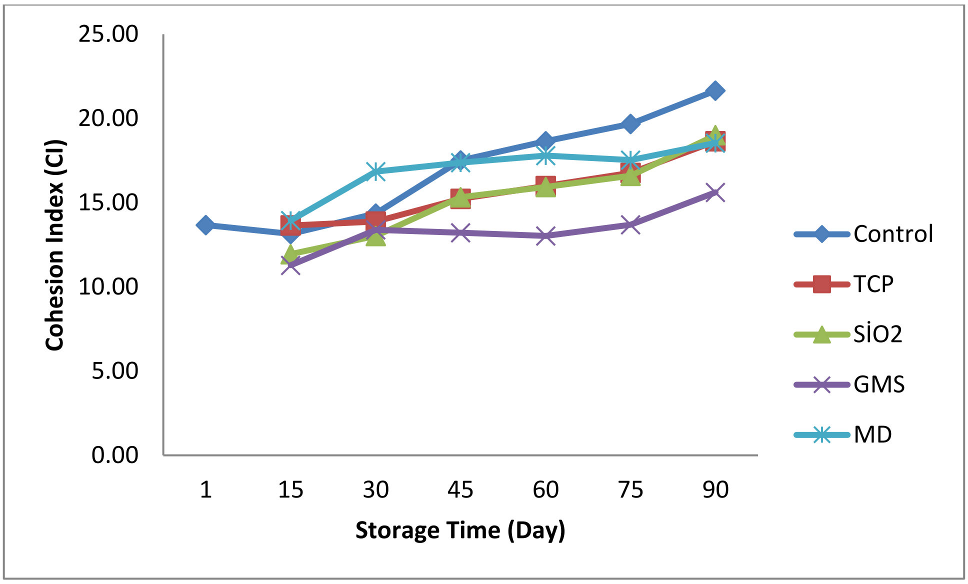

Figure 1. Cohesion index values of coffee samples during storage time
SiO_2 : Silicon dioxide, MD: maltodextrin, GMS: glycerol monostearate, TCP: tricalcium phosphate

Caking profiles

Due to their structure, powder products can come together and become lumps over time. This situation, which results in irregular collection and loss of properties, can be defined as caking (Mercan et al., 2018). Additionally, caking may have effects such as changing product properties and completely deteriorating the product in powder groups (Aguilera et al., 1995).

Caking problem is important in the storage period and packaging material selection of all powder products. Even the selection of the countries to which the manufactured products will be sent may vary due to the caking problem in powder product groups. Cake products are not preferred by consumers because they have lost their properties and are non-homogeneous powder mixtures. The mean cake strength values of instant coffee samples measured for this purpose are given in Table 1. When the table is examined, it is striking that the maltodextrin sample never formed cake during the storage period, and again, no cake was formed in the GMS sample during the 75th day of storage. On the other hand, there is a caking value in the control group coffee samples that increases during storage. In addition, although the addition of SiO_2 and TCP to coffee formulations significantly reduced the caking values, an increase in the caking values was observed in these samples during storage. Anticaking agents bind more moisture to the product in powder mixtures, create a physical barrier within the product, eliminate friction or limit the interaction between powders, thus preventing the formation of caking (Barbosa-Cánovas et al., 2005). It is thought that the agents used in the study prevent caking by keeping the components in the coffee mixture separate.

Powder speed flow dependence

The PFSD test expresses the flow stability of powder mixtures and how the powder changes during analysis (Jan et al., 2015). Compaction coefficient values of ready-to-drink coffee samples depending on increasing test speeds are shown in Figure 2. The 15th and 90th day values are shown graphically to serve as an example in storage time. As the compression rate increased, the compression coefficient of Control, GMS and MD samples decreased on the 15th day, while no significant change was observed in TCP and SiO_2 samples. On the 90th day, with increasing test speed, the compression coefficient values of GMS and MD samples decreased again, while TCP remained constant, control and SiO_2 showed a general increasing trend. Changes in compression coefficient values were detected in SiO_2 and control samples with increasing storage time.

Table 1. Mean cake strength (g mm) values of coffee samples

Storage Day	1	15	30	45	60	75	90
Control	93.90 ± 5.85^{Ea}	116.03 ± 3.57^{Da}	122.49 ± 2.37^{Ca}	124.59 ± 3.40^{Ca}	153.46 ± 2.54^{Ba}	158.06 ± 1.46^{Aa}	159.63 ± 0.18^{Aa}
TCP	-	CAKE NOT FORMED	27.17 ± 0.69^{Cc}	33.88 ± 0.27^{Bc}	34.27 ± 0.21^{Bc}	34.71 ± 0.54^{Bc}	47.68 ± 0.12^{Ac}
SiO_2	-	36.15 ± 2.21^{Eb}	38.44 ± 1.21^{Db}	40.09 ± 0.51^{Cb}	40.11 ± 0.83^{Cb}	43.68 ± 0.14^{Bb}	49.10 ± 1.45^{Ab}
GMS	-	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	30.667 ± 2.44^{Ad}
MD	-	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED	CAKE NOT FORMED

Means followed by the same small letter in column and capital letter in row do not differ by Tukey's test ($p > 0.05$). Data are mean values of triplicate samples ± with standard deviation. SiO_2 : Silicon dioxide, MD: maltodextrin, GMS: glycerol monostearate, TCP: tricalcium phosphate

Table 2. Flow stability of coffee samples

Storage Day	**1**	**15**	**30**	**45**	**60**	**75**	**90**
Control	0.83 ± 0.03^{Ca}	0.90 ± 0.04^{Bc}	0.93 ± 0.03^{Bb}	0.99 ± 0.03^{Ab}	1.03 ± 0.04^{Aa}	0.98 ± 0.04^{Ab}	1.02 ± 0.03^{Aa}
TCP	-	1.01 ± 0.04^{Ab}	0.98 ± 0.04^{Aa}	1.03 ± 0.01^{Ab}	0.98 ± 0.02^{Ab}	1.01 ± 0.02^{Ab}	1.00 ± 0.02^{Aa}
SiO_2	-	0.97 ± 0.07^{Bb}	1.00 ± 0.02^{Aa}	1.10 ± 0.11^{Aa}	1.04 ± 0.03^{Aa}	1.07 ± 0.01^{Aa}	1.03 ± 0.06^{Aa}
GMS	-	0.88 ± 0.07^{Bd}	1.00 ± 0.10^{Aa}	1.01 ± 0.10^{Ab}	1.06 ± 0.09^{Aa}	0.98 ± 0.01^{Ab}	0.96 ± 0.69^{Ab}
MD	-	1.10 ± 0.10^{Aa}	0.94 ± 0.11^{Bb}	0.98 ± 0.04^{Bb}	0.98 ± 0.07^{Bb}	0.98 ± 0.04^{Bb}	0.96 ± 0.04^{Bb}

Means followed by the same small letter in column and capital letter in row do not differ by Tukey's test ($p > 0.05$). Data are mean values of triplicate samples ± with standard deviation. SiO_2 : Silicon dioxide, MD: maltodextrin, GMS: glycerol monostearate, TCP: tricalcium phosphate

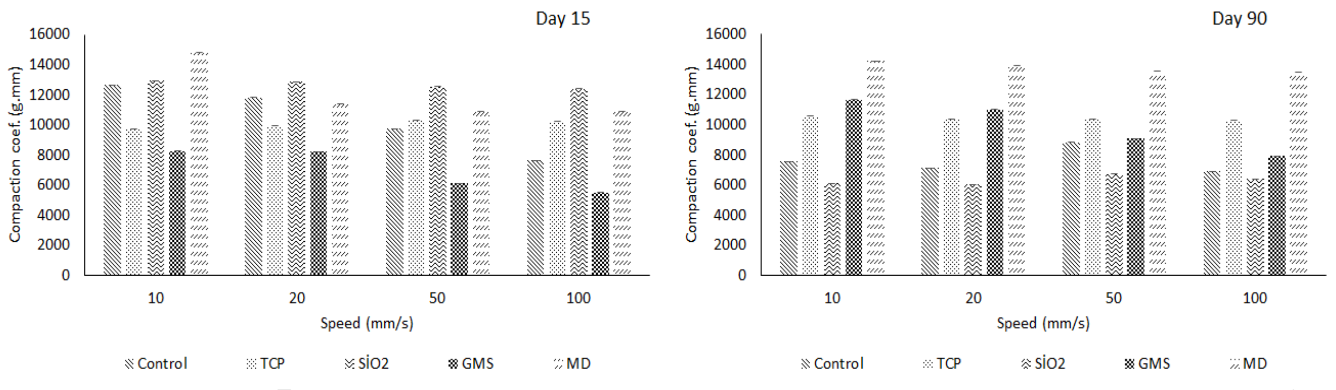

Figure 2. PFSD compaction coefficient trends of the powdered coffee samples

The flow stability of coffee samples is shown in Table 2. An increase in flow stability values was generally observed during the storage period of ready to drink coffee blends. However, flow stability values decreased in instant coffees with maltodextrin added. Especially in coffees that are referred to as control and do not contain any anti-caking agent, the flow stability values started with 0.83 and were determined as 1.02 at the end of storage. On the other hand, at the end of the storage period, the lowest flow stability value was determined as 0.96 in instant coffees with GMS and MD additions. Additionally, no difference could be detected in the stability values of control, TCP and SiO_2 added coffee samples as a result of storage ($p > 0.05$). If the flow stability value is equal to 1, it is stated that the form of the powder product does not change. If it is less than or greater than 1, it is explained as the powder changing during the analysis (Benkovic and Bauman, 2009).

Scanned electron microscopy (SEM)

Figure 3 shows SEM images of instant coffee mixtures. SEM images are evaluated to show the changes of powder samples during storage processes. SEM images were obtained after the 1st and 90th days of storage for coffee samples without any anti-caking agent, and after the 90th day of storage for coffee samples containing anti-caking agent. SEM images of the samples are shown at 100x and 500x magnification. As a result of the evaluation, 500x magnification images of the samples prepared with maltodextrin (MD) anti-caking agent show that there is no caking and the ingredients in the instant coffee mixture are present separately. Among the anti-caking agents, it can be said that MD is the best additive in instant coffee formulation.

Anti-caking agents prevent caking by different mechanisms such as water binding and barrier formation. For example, it is known that silicon dioxide coats the main product due to its small particle size and thus prevents caking (Nurhadi ve Roos, 2017). When the SEM images are examined, it is thought that anticaking agents surround the particles of the components in the coffee mixture and prevent them from coming together.

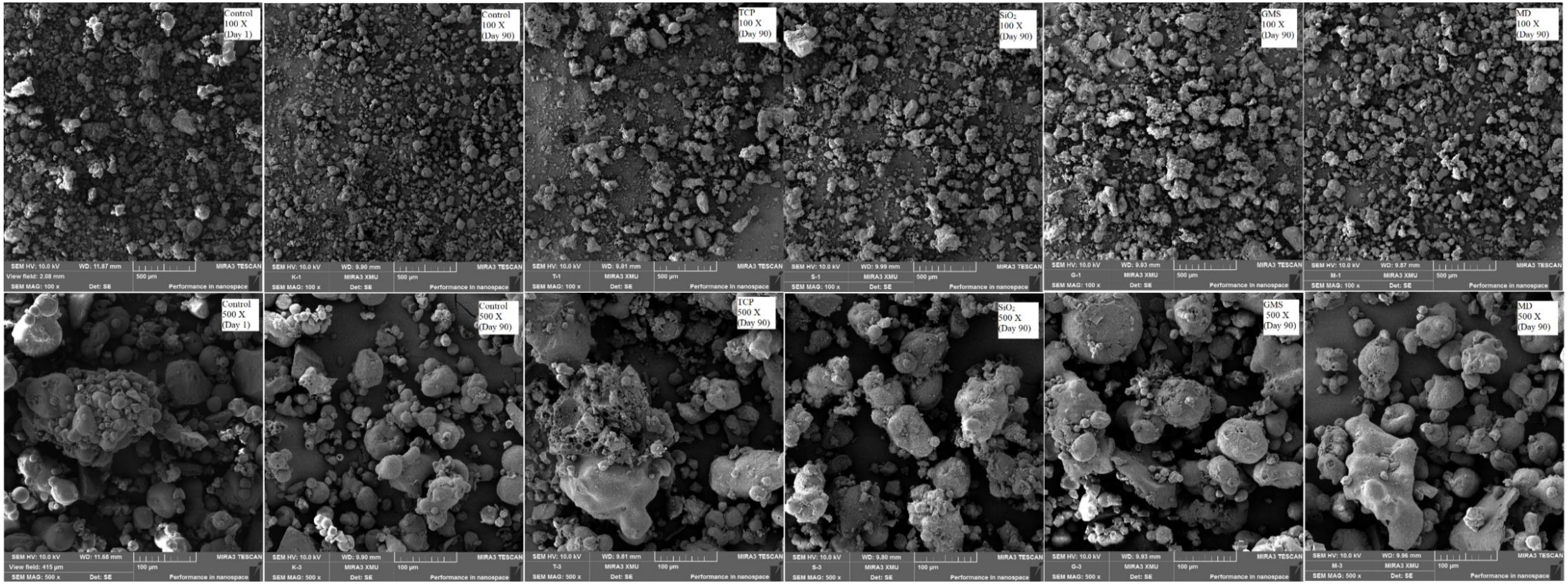

Figure 3. SEM images of coffee samples

Conclusion

In this study, different anti-caking agents were added to ready-to-drink coffee samples and the changes in the powder product form during the storage period were determined by powder flow and SEM imaging. The problems of agglomeration and caking of powdered products effect the choice of packaging material, determination of storage processes and even determination of transfer routes for countries where they can be sent abroad. For this reason, the outputs of the study are considered important for ready-to-drink coffee samples that are consumed quite frequently. As a result of the study, it was determined that the caking values of the control coffee samples without any anti-caking agent increased during the storage process. On the other hand, it was determined that agents such as maltodextrin and glycerol monostearate did not cause caking and other additives used significantly reduced the caking values. In addition, SEM images were used to determine the agglomeration and caking of powders in the powder product and the visual results were similar to the powder flow values. It was observed that the anti-caking agents selected for the study were very effective during the storage period. It was observed that the anti-caking agents selected for the study were quite effective during the storage period. After this stage, new studies can be conducted with mixture forms to determine the synergistic effects of anti-caking agents, different anti-caking agents can be tried in the coffee sample, and the effects of the agents used within the scope of the study can be tested in different powder product groups to investigate their effects. Because of our study is a very new research in terms of literature, we believe that there is a need for a lot of new research in this field regarding powder product groups.

Conflict of interest: The authors declared no potential conflicts of interest regarding the publication of this article.

Authors' Contribution: Duygu ASLAN TÜRKER and Meryem GÖKSEL SARAÇ were responsible for selection of the research topic, conducting an experiment, data collection and analysis, writing and submitting the manuscript. Mahmut DOĞAN reviewed the manuscript.

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