International Journal of Chemical Studies

P-ISSN: 2349–8528
E-ISSN: 2321–4902
IJCS 2019; 7(1): 1714-1718

Received: 12-11-2018
Accepted: 15-12-2018

Gaikwad SA
MIT College of Food Technology, Loni Kalbhor, Pune, Maharashtra, India

Kulthe AA
MIT College of Food Technology, Loni Kalbhor, Pune, Maharashtra, India

Suthar TR
MIT College of Food Technology, Loni Kalbhor, Pune, Maharashtra, India

Correspondence
Kulthe AA
MIT College of Food Technology, Loni Kalbhor, Pune, Maharashtra, India

Characterization of flavoured sweet water balls prepared by basic spherification technique

Gaikwad SA, Kulthe AA and Suthar TR

Abstract
Spherification, an innovative technique of molecular gastronomy which is a recent development in the field of food science and technology, can be applied to create high quality foods having high sensory properties and degree of consumer acceptability and satisfaction. It is an ideal technique for obtaining semi-solid spheres with very thin membrane coating that is almost imperceptible in the mouth. The present study involved the preparation of flavoured sweet water balls with sodium alginate by basic spherification technique. The prepared balls were evaluated for their change in physical properties and drying behaviour in accordance to different concentration of sodium alginate solution (1, 2, 3, 4 and 5%, w/v) and gelling time (3, 6, 9 and 12 min). Significant increase in the diameter and gel membrane thickness of alginate balls was observed. Variation in sodium alginate concentration and gelling time also affected the rate of moisture loss during drying. The flavoured sweet water balls containing 25% sugar and 3% sodium alginate with 6 min gelling time were found to be more acceptable on the basis of sensory evaluation.

Keywords: Spherification, sodium alginate, calcium chloride, physical properties, drying behaviour, sensory quality

1. Introduction
Flavoured sweet water balls are gelatinous, double-membrane spheres made by pouring flavoured sweet water into separate solutions of calcium salt and sodium alginate. Spherification, an old technique in the world of modernist cuisine, was pioneered at El Bulli in 2003 and is a cornerstone in experimental kitchens across the world (Vega and Castells, 2012) [16]. The idea of exploiting spherification-like techniques for food production was first patented in Britain in 1942 by William Peschardt, a food scientist working for the firm Unilever. Since then, the technique-using ions to trigger a gelling process-has found a variety of uses in the food industry.
Spherification is a modern technique that involves creating semi-solid spheres with thin membranes out of liquids, which are filled with a non-gelled liquid. As a result of this, a burst-in-the-mouth effect is achieved with the liquid. It is the process of trapping a liquid inside of a gelled sphere. Both flavour and texture is enhanced with the spherification technique. Spheres can be made in various sizes as well as firmness.
Spherification is the shaping of liquid into edible spheres in calcium-alginate capsules by application of the technique of jellification. It is a controlled jellification of a liquid that is based on the crosslinking reaction between calcium chloride and sodium alginate. The spheres formed by the spherification process, have liquid centres covered within a gel capsule. This makes it possible to encase liquids within the solid spheres. There are two main types of spherification techniques *viz.* the basic and reverse techniques. The reverse or external technique utilizes a calcium source added to edible liquid and dropped in a gelling solution and water bath like that of sodium alginate bath. In basic spherification, gelling solutions like sodium alginate are mixed directly with the chosen liquid and dropped in calcium chloride and water bath to form a thin gel shell. In this version, the spheres are easily breakable and should be consumed immediately.
Alginate is a naturally occurring anionic polymer typically obtained from brown seaweed (Mabeau and Fleurence, 1993) [5], and has been extensively investigated and used for many biomedical applications (Silva *et al.*, 2006) [12], due to its biocompatibility, low toxicity, relatively low cost, and mild gelation by addition of divalent cations such as Ca^{2+}. Sodium alginate is a linear unbranched polysaccharide which consist of 1,4 linked β-D-mannuronic

and 1,4 α-L-guluronic acid residue (Smardel *et al*., 2008) [13]. Alginates linked have an affinity for alkaline earth metals and the affinity increases in the order $Mg^{2+}<Ca^{2+}<Sr^{2+}<Ba^{2+}$ (Kohn, 1975) [3]. In the absence of divalent ions (i.e., Ca^{2+}, Mg^{2+}) alginates only enhance the viscosity; however, when in the presence of divalent ions, especially calcium, they form strong gels. In the deprotonated state, at pH levels less than 5, the regions of the copolymer concentrated in guluronic acid (i.e., depleted in mannuronic acid) are able to strongly interact with calcium forming a divalent salt bridge between alginate polymers (Martinsen *et al.*, 1989) [8]. Therefore, the gel forming properties of alginates are derived from their capacity to bind a large number of divalent ions and the gel strength is correlated with the proportion and length of the guluronic acid blocks (G-blocks) in their polymeric chains (Mancini and McHugh, 2000) [6]. The salt bridges that are responsible for the skin formation (outer gel layer) of alginate spheres have been described using the "egg-box" model by Lee and Rogers (2012) [4].

Sodium alginate performs various functional properties such as it acts as a thickener, gelling agent, binder and stabilizer in food industries. It is used for encapsulation of volatile agents for drug delivery or flavour entrapment, due to its property of forming ionotropic gelation with calcium ion under mild conditions (Mandal *et al*., 2010) [7]. The various applications of sodium alginate include use of the same as stabilizer for ice cream, yogurt, cream, and cheese. It acts as a thickener and emulsifier for salad, pudding, jam, tomato juice, and canned products.

The gelling agents used in spherification only gel in the presence of certain ions, such as calcium or potassium. Although alginate gels, more specifically partially gelled orbs using alginates and a calcium source, were introduced in 2003 the technology has been around for decades in the food industry, which first used alginates to restructure red peppers for manufacturing pimentos in olives. Unlike most edible gels, which are solid throughout, alginate spheres typically contain a physical outer gel membrane with a liquid core. Various calcium salts like chlorides, lactates, gluconoates, etc. are used for jellification process. Typically calcium chloride is commonly used because it reacts rapidly with the alginate forming the divalent salt bridges and gels. Calcium chloride rapidly dissociates when added to solution because of its high solubility, making it an attractive calcium source for spherification (Lee and Rogers, 2012) [4].

Challenges related to spherification include the choice of the correct concentration of sodium alginate, gelling time and concentration of sugar syrup. The present investigation was carried out to standardize the process for preparation of flavoured sweet water balls using sodium alginate, calcium chloride and sugar syrup by basic spherification technique and to examine the physical and sensory properties of the resultant product as affected by sodium alginate concentration, gelling time and sugar syrup concentration.

Materials and Methods

Materials

Sodium alginate ($C_6H_7O_6Na$, Mol. Wt. 85000) and calcium chloride dehydrate AR grade used in present investigation were supplied by M/s. Setlab India, Pune. Sugar, mango flavour and yellow colour were procured from the local market.

Preparation of flavoured sweet water balls

Sugar syrup and, sodium alginate were taken in various concentrations with constant level of calcium chloride (3% w/v) at different gelling time intervals in order to study the effect of the same on size, shape and wall thickness of balls. For preparation of flavoured sweet water balls (Fig. 1), different concentrations of sugar syrup (10, 15, 20 and 25%) were incorporated in sodium alginate solutions (1, 2, 3, 4 and 5%, w/v). Colour (yellow) and flavour (mango) were added to the resulting dispersion and then dropped slowly with help of scoop into 3% (w/v) aqueous calcium chloride solution followed by stirring. After stirring for different time intervals (3, 6, 9 and 12 minutes), the formed balls were separated by filtration, washed with distilled water and dried at ambient temperature (Menon and Sajeeth, 2013) [9]. The prepared balls were preserved for further evaluation of physical and sensory properties.

Evaluation of alginate balls

Gelling time

Gelling time is the time required by the alginate beads to form gel. The alginate balls were prepared by dropping the dispersion into calcium chloride solution, allowed to harden for different time periods (3, 6, 9 and 12 min) in order to examine the time taken to form gel (Smardel *et al*., 2008) [13].

Physical quality evaluation of alginate balls

The alginate balls were evaluated for their diameter and gel membrane thickness. The diameter and gel membrane thickness of the balls were measured by using vernier caliper. For gel membrane thickness, the balls were cut to remove the liquid inside, while the coating membrane was washes with water and its thickness was measured using a vernier caliper (Sherina *et al*., 2012) [11].

Drying behaviour of alginate balls

The prepared balls with different concentration of alginate liquid (1.0%, 2.0%, 3.0%, 4.0% and 5.0% (w/v)) with gelling time as 6 minutes were kept in hot air oven at 50 °C. The weight of balls was measured after every half an hour. The measurements were continuously done until the constant weight is observed (Sherina *et al*., 2012) [11].

Sensory evaluation

The flavoured sweet water balls were evaluated for sensory attributes by a panel of 15 semi-trained judges, using a 9 point Hedonic scale system for different parameters like colour and appearance, flavour, texture, taste and overall acceptability. The mean values of 15 semi-trained judges were considered for evaluating the quality.

Statistical analysis

The data obtained was analyzed statistically to determine statistical significance of treatments. The figures then were averaged. The data obtained was analyzed statistically using standard methods given by Snedecor and Cochran (1987) [14] and by Duncan's multiple range test with the probability $p \leq 0.05$ (Duncan, 1955) [1]

Result and Discussion

Spherification was introduced by the famous executive chef Ferran Adria, as a technique of molecular gastronomy in 2003s. It is a process in which calcium chloride and alginate react together at a controlled rate, thus resulting in entrapment of liquid in a thin membrane of gel. In the present study, the edible balls prepared by basic spherification technique with variation in sodium alginate concentration (1, 2, 3, 4, and 5%, w/v) and varied gelling time (3, 6, 9, 12 min) were prepared.

For the preparation of alginate balls, a measured quantity of alginate solution was poured (in horizontal position) using a scoop of desired size into the calcium chloride bath with the minimum distance between the scoop and bath to create a perfectly round shape. The alginate balls were carefully removed from the calcium chloride bath after obtaining desired texture (3 to 12 min) and rinsed with clean water. The prepared alginate balls were evaluated for their physical properties as affected by different concentration of sodium alginate solution and gelling time. The results obtained are discussed as below.

Effect of sodium alginate concentration and gelling time on physical properties of alginate balls

The effect of different concentrations of sodium alginate (1, 2, 3, 4, and 5%, w/v) and varied gelling time (3, 6, 9, 12 min) on the physical properties is presented in table 1. It was observed that the physical properties *viz.* diameter and gel membrane thickness of alginate balls significantly varied with respect to sodium alginate concentration and gelling time.

The diameter of the alginate balls significantly increased ($p<0.5$) with increase in both concentration of sodium alginate solution as well as gelling time. With increase in sodium alginate concentration from 1 to 5% (w/v), the diameters of alginate balls increased from 3.08 to 4.16 cm, 3.23 to 4.58 cm, 3.63 to 4.72 cm and 3.85 to 4.86 cm in respect to the gelling time of 3, 6, 9 and 12 min. The increase in diameter and thickness of gel membrane of alginate balls may be attributed to the rate of gelation caused by diffusion of calcium ions into alginate solution which is directly proportional to the concentration of sodium alginate and gelling time (Lee and Rogers, 2012) [4].

The thickness of gel membrane of the alginate balls significantly increased ($p<0.5$) from 1.32 to 1.78 cm, 1.43 to 1.89 cm, 1.47 to 1.94 cm and 1.54 to 2.04 cm with increase in concentration of sodium alginate solution (from 1 to 5%, w/v) and gelling time (from 3 to 12 min). It was observed that as the alginate solution was poured into the calcium chloride bath; rapid gelation was induced resulting in instant gel membrane formation around the alginate solution that proceeded very rapidly. The gel membrane was formed due to cross liking reaction between guluronic acid blocks of sodium alginate and calcium ions, resulting in egg-box shaped three dimensional matrix ultimately leading to gelation (Martinsen *et al*., 1989) [8]. The degree of matrix formation depends on the concentration of sodium alginate and calcium ions. Thus more dense matrix (gel membrane) formed with high concentration of sodium alginate (Park *et al*., 1997) [10]. Also, as the gelling time increased, the rate of calcium ion cross linking with alginate solution also increased. The rapid cross linking caused increased development of the elastic component of the gel forming a dense matrix, thus increasing the thickness of gel membrane around the alginate balls (Mancini and McHugh, 2000) [6]. Similar results were observed by Yong *et al.*, 2011 [17] for the effect of sodium alginate concentration on physical properties of persimmon calcium alginate beads.

Effect of sodium alginate concentration and gelling time on drying behaviour of alginate balls

The data on drying behaviour of alginate balls affected by variation in sodium alginate concentration and gelling time is displayed in figure 2. All the alginate ball samples showed more or less similar pattern for decrease in their weights during drying period. The decrease in weight was attributed to the loss of moisture from the alginate balls during drying. Drying significantly changed the shape of alginate balls causing shrinkage of balls and wrinkles on the surface of dried samples (Smardel *et al*., 2008) [13]. The drying rate was observed to be high in case of samples containing low sodium alginate concentration and lower gelling time. The loss of moisture decreased with increase in sodium alginate concentration (1 to 5% w/v) and gelling time (3 to 12 min). This variation in drying rate may be attributed to the thickness of gel membrane in alginate balls which formed a barrier for mass transfer during drying. It was observed that alginate balls with 3% sodium alginate and 6 min gelling time possessed best characteristics.

Sensory evaluation of flavoured sweet water balls

The flavoured sweet water balls (Fig. 1) containing 3% sodium alginate solution and varied concentration of sugar syrup (10, 15, 20 and 25%) with gelling time of 6 min were prepared by similar method as discussed above. To enhance the appeal of the balls, yellow colour and mango flavour were added. The prepared flavoured sweet water balls were evaluated for sensory attributes *viz*. colour and appearance, flavour, texture, taste and overall acceptability. Table 2 depicts the data on sensory evaluation of flavoured sweet water balls. The data revealed that samples containing 25% sugar syrup secured highest scores for texture (8.56), taste (8.78) and overall acceptability (8.16) in comparison to other samples. Samples containing 10% sugar syrup secured least scores for sensory parameters. The difference in taste scores may be attributed to high sweetness of samples with 25% sugar syrup. The concentration of sugar syrup also affect the strength and water holding capacity of flavoured sweet water balls, thus creating difference in their texture scores. Similar results were observed by Kaur *et al*., 2018 [2], Yong *et al*., 2011[17] and Tang *et al*., 2001 [15].

Table 1: Effect of sodium alginate concentration and gelling time on the physical properties of alginate solution

Treatments	Sodium Alginate (%, w/v)	Gelling Time (min)	Ball Diameter (cm)	Gel Membrane Thickness (cm)
S_1G_1	1.0	3	3.08 (±0.06)^{d}	1.32 (±0.02)^{a}
S_2G_1	2.0	3	3.27 (±0.12)^{d}	1.44 (±0.05)^{d}
S_3G_1	3.0	3	3.62 (±0.14)^{c}	1.52 (±0.03)^{cd}
S_4G_1	4.0	3	3.95 (±0.08)^{b}	1.63 (±0.04)^{c}
S_5G_1	5.0	3	4.16 (±0.05)^{b}	1.78 (±0.04)^{b}
S_1G_2	1.0	6	3.23 (±0.11)^{d}	1.43 (±0.03)^{d}
S_2G_2	2.0	6	3.54 (±0.12)^{cd}	1.58 (±0.05)^{c}
S_3G_2	3.0	6	3.87 (±0.18)^{c}	1.68 (±0.03)^{bc}
S_4G_2	4.0	6	4.24 (±0.20)^{b}	1.74 (±0.04)^{b}
S_5G_2	5.0	6	4.58 (±0.16)^{a}	1.89 (±0.04)^{a}
S_1G_3	1.0	9	3.63 (±0.10)^{c}	1.47 (±0.05)^{d}
S_2G_3	2.0	9	3.84 (±0.17)^{c}	1.58 (±0.05)^{c}

S_3G_3	3.0	9	4.07 (±0.12)^b	1.71 (±0.04)^b
S_4G_3	4.0	9	4.35 (±0.24)^b	1.86 (±0.06)^{ab}
S_5G_3	5.0	9	4.72 (±0.06)^a	1.94 (±0.02)^a
S_1G_4	1.0	12	3.85 (±0.11)^c	1.54 (±0.04)^c
S_2G_4	2.0	12	4.22 (±0.12)^b	1.63 (±0.03)^c
S_3G_4	3.0	12	4.45 (±0.25)^{ab}	1.76 (±0.06)^b
S_4G_4	4.0	12	4.73 (±0.10)^a	1.98 (±0.04)^a
S_5G_4	5.0	12	4.86 (± 0.12)^a	2.04 (±0.03)^a

1. Each value is the average of three determinations
2. Values are means (± standard deviation).

Means not sharing a common superscript letter(s) in a column are significantly different at $p \leq 0.05$ as assessed by Duncan's multiple-range test.

Table 2: Sensory evaluation of flavoured sweet water balls

Sugar Syrup Concentration	Colour & Appearance	Flavour	Texture	Taste	Overall Acceptability
10%	7.14 (±0.03)^c	7.07 (±0.04)^c	8.02 (±0.06)^d	8.12 (±0.05)^d	7.59 (±0.08)^d
15%	7.35 (±0.05)^b	7.15 (±0.03)^{bc}	8.14 (±0.05)^c	8.31 (±0.05)^c	7.74 (±0.05)^c
20%	7.87 (±0.02)^a	7.21 (±0.05)^{ab}	8.33 (±0.08)^b	8.50 (±0.07)^b	7.98 (±0.06)^b
25%	7.92 (±0.01)^a	7.36 (±0.02)^a	8.56 (±0.07)^a	8.78 (±0.08)^a	8.16 (±0.05)^a

1. Each value is the average of fifteen determinations
2. Values are means (± standard deviation).
3. Means not sharing a common superscript letter(s) in a column are significantly different at $p \leq 0.05$ as assessed by Duncan's multiple-range test.

Fig 1: Flavoured sweet water balls

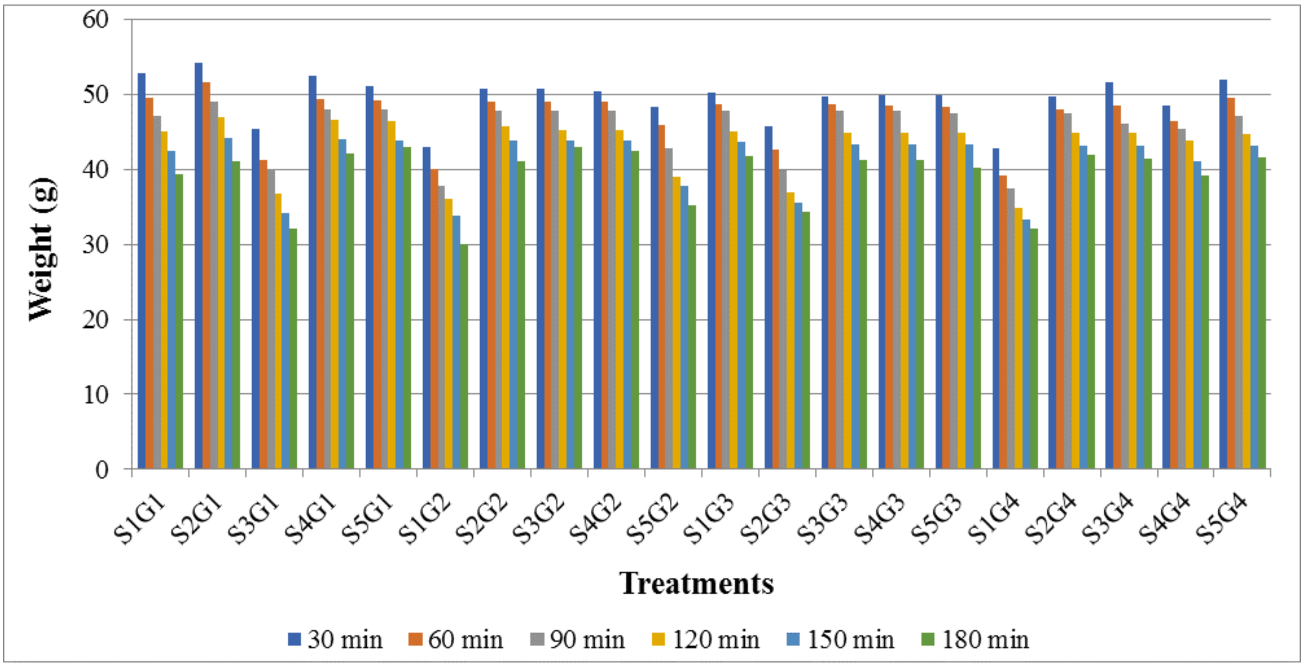

Fig 2: Effect of sodium alginate concentration and gelling time on drying behaviour of alginate balls

Conclusion

The results show that flavoured sweet water balls can be prepared with sugar, sodium alginate and calcium chloride by basic spherification technique. The physical properties of alginate balls *viz*. diameter and gel membrane thickness are significantly affected by the sodium alginate concentration and gelling time. The rate of drying reduced with increase in sodium alginate concentration and gelling time. It was observed that alginate balls with 3% sodium alginate and 6 min gelling time possessed best characteristics. Sensory evaluation of flavoured sweet water balls revealed that samples with 25% sugar syrup had highest consumer acceptability.

References

1. Duncan BD. Multiple-range and multiple F-test. Biometrics. 1955; 11:1-42.
2. Kaur N, Singh B, Sharma S. Hydrogel for potential food application: Effect of sodium alginate and calcium chloride on physical and morphological properties. The Pharma Innovation Journal. 2018; 7(7):142-148.
3. Kohn R. Ion binding on polyuronates-alginate and pectin. Pure and Applied Chemistry. 1975; 42:371-397.
4. Lee P, Rogers MA. Effect of calcium source and exposure time on basic cavier spherification using sodium alginate. International Journal of Gastronomy and Food Science. 2012; 1(2):96-100.
5. Mabeau S, Fleurence J. Seaweed in food products: Biochemical and nutritional aspects. Trends in Food Science and Technology. 1993; 4:103-107.
6. Mancini F, McHugh TH. Fruit-alginate interactions in novel restructured products. Nahrung. 2000; 44:152-157.
7. Mandal S, Kumar SS, Krishnamoorthy B, Basu SK. Development and evaluation of calcium alginate beads prepared by sequential and simultaneous methods. Brazilian Journal of Pharmaceutical Science. 2010; 46:785-793.
8. Martinsen A, Skjak Braek G, Smidsrod O. Alginate as immobilization material: I. Correlation between chemical and physical properties of alginate beads. Biotechnology and Bioengineering. 1989; 33:79-89.
9. Menon TV, Sajeeth CI. Formulation and evaluation of sustained release sodium alginate microbeads of carvedilol. International Journal of Pharmaceutical Technology Research. 2013; 5:746-753.
10. Park JS, Kim DK, Lee KH, Doh DS. The diffusion characteristics of colute into the Ca-alginate gel bead. Hwahak Konghak. 1997; 35(1):41-45.
11. Sherina VM, Santhi K, Sajeeth CI. Formulation and Evaluation of Sodium alginate microbeads as a carrier for the controlled release of Nifedipine. International Journal of Pharmaceutical Chemical Science. 2012; 1:699-710.
12. Silva CM, Ribeiro AJ, Figueiredo IV, Goncalves AR, Veiga FX. Alginate microspheres prepared by internal gelation: Development and effect on insulin stability. International Journal of Pharmaceuttics. 2006; 311(1-2):1-10.
13. Smardel P, Bogataj M, Mrhar A. The influence of selected parameters on the size and shape of alginate beads prepared by ionotropic gelation. Science Pharmaceutics. 2008; 76:77-89.
14. Snedecor GW, Cochran WG. Statistical Methods (17th Edn.) Ames, IA.: The Lowa State University Press, 1987, 221-222.
15. Tang J, Mao R, Tung MA, Swanson BG. Gelling temperature, gel clarity and texture of gellan gels containing fructose or sucrose, Carbohydrate Polymer. 2001; 44:197-209.
16. Vega C, Castells P. Spherification. In: Vega, C., Ubbink, J., Van der Linden, E. (Eds.). The Kitchen as the Laboratory. Columbian University Press, New York, 2012, 25-32.
17. Yong DH, Song M, Yoon HH. Effects of sodium alginate concentration on physical and sensory characteristics of persimmon calcium alginate beads. The Korean Journal of Food and Cookery Science. 2011; 27(5):497-505.