



Development of Edible Water Bubbles by Spherification

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Abstract: Use of single use plastic bottles has been constantly increasing with its convenience being a major advantage. But this increasing use of plastics have also contributed to increased waste production, as millions of bottles are being thrown away without proper recycling. Around 1 million of such bottles are being purchased every minute around the world and are used in various events. A natural solution to reduce plastic bottle usage was first brought up by the Skipping Rocks Lab in the form of 'ooho! Bubbles'. The concept behind this was to encapsulate the water within an edible seaweed membrane, formed by implementing a popular molecular gastronomy process known as Spherification. The membrane is formed by the polymerization of Sodium alginate in the presence of Calcium ions, each dissolved in water individually. The membrane can be consumed along with the encapsulated water, or thrown away to be naturally decomposed, thus causing no pollution. This article is based on the process of Spherification and its types, the conditions required for spherification and the various physical properties of the bubble formed. Trials have been made using different liquids and in different salt concentrations, to replicate the process and analyse the results. A survey has been conducted online to find out consumer awareness on single use plastics, effects of bottled water and the acceptance of edible packaging.

Index terms - Calcium chloride, Calcium lactate, encapsulation, gel membrane, Sodium alginate, spherification

I. INTRODUCTION

Single-use plastics (SUPs) are made primarily from fossil fuel-based chemicals and are meant to be disposed of right after use. Plastic is found in an almost infinite number of applications, while packaging contributes the most to the demand for plastic. Around 300 million plenty of plastic is produced annually worldwide, half which is for single-use items (Courtney Lindwall, 2020). Due to their improper recycling, SUPs are causing an increase in pollution.

"ooho! Bubbles" was the first product that was developed based on the concept of "edible water bubbles", by the Skipping Rocks Lab. The bubble is formed as an edible seaweed membrane, made from alginates and calcium solutions, by a method known as spherification (Amy Frearson, 2019). The main aim of these edible bubbles is to reduce the use of single use plastic bottles, and in turn reduce pollution.

Spherification is a popular and innovative process of molecular gastronomy, also known as an encapsulation technique that involves the creation of semi-solid spheres with thin membranes out of liquids, filled with a non-gelled liquid core. The sphere membrane, if not consumed, degrades naturally in few weeks, thus causing no pollution.

In this work, trials to develop edible water bubbles using the spherification process was done, and the effect of alginate concentration and gelation period was observed when done using different liquids. A survey was conducted online, to understand consumer's awareness about single use plastic and acceptance of edible packaging in India.

II. SPHERIFICATION

Spherification is known to be one among the popular processes of Molecular Gastronomy (MG) and English food scientist William J. S. Peschardt is being credited for the invention of spherification, who patented the technique in the 1940s (Bethany Halford, 2014).

Molecular gastronomy (MG) can be defined as the scientific activity consisting in looking for the mechanisms of phenomena occurring during dish preparation and consumption. The Spanish chef Ferran Adrià is known for development of the technique

called spherification, whose basis is the encapsulation using sodium alginate spheres. Spherification was also looked into for use in liquid foods of different nature, by modelling charged, hydrophilic, and hydrophobic compounds. (Nicola and Diego, 2015)

Spherification is an old technique in the modernist cuisine world, which was pioneered at El Bulli in 2003. As an innovative technique of molecular gastronomy, spherification can be applied to create foods with high quality, sensory properties and degree of consumer satisfaction and acceptance. Spherification involves the creation of semi-solid spheres with thin membranes out of liquids, filled with a non-gelled liquid, to attain a bursting-the-mouth effect. i.e., it is simply the process of trapping a liquid inside of a gelled sphere. Spheres made by this technique can be in various sizes and firmness. Flavour and texture can be enhanced by the technique of spherification. The choice of correct concentration of sodium alginate and gelling time are mentioned as some of the challenges in this technique. The physical properties of alginate balls such as its diameter and gel membrane thickness are affected in a wide range by the sodium alginate concentration and gelling time. (Gaikwad SA *et al*, 2019)

Direct or reverse spherification process is followed generally, and these methods are based on the effect that gelling mixtures do not set, unless charged molecules are made present. In the direct spherification approach, a water solution in which the beverage or liquefied food together with the gelling agent (usually sodium alginate) is added gently to a second bath of calcium gluconate, that contains the missing ions. When sodium solution is dropped in the calcium bath, an immediate gelification process happens, creating small gel capsules with a liquid core or chewy beads. While in reverse spherification, the calcium lactate or other sources of calcium ions is added to the sodium liquid. The round shape of the droplets is attributed to its surface tension. (Nicola Caporaso, 2021). Frozen reverse spherification involves the extra step of pre-freezing liquid foods with a calcium content in a spherical mold, before immersing them in a sodium alginate bath. (Yan Ling Low and Liew Phing Pui, 2020)

Structure of Alginates comprises of unbranched copolymers of 1,4-linked- β -D-mannuronic acid and α -L-guluronic acid, and are found with an affinity for alkaline earth metals. In the presence of divalent ions, (calcium ions Ca^{2+} in specific), sodium alginate tends to form strong gels. This property of alginates is due to their ability to bind a high number of divalent ions. The gel strength is related with the proportion and length of the guluronic acid blocks (G-blocks) in the polymeric chains of alginates (P.Lee and M.A.Rogers, 2012) Sodium alginate can form a gel in room temperature, though stable gel can be formed in a temperature range of 0 - 100°C. This gel can be preserved more completely when kept immersed in a water bath. (Pumpho and Puechkamutr, 2014)

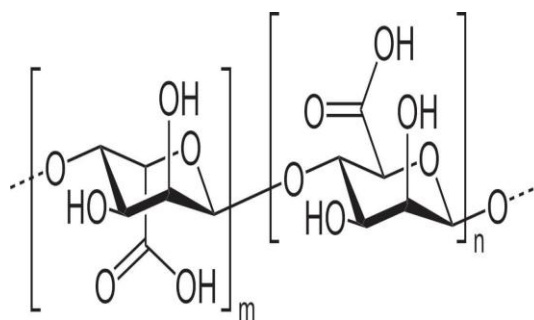
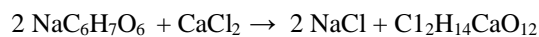


Fig. 1 Molecular structure of Sodium alginate

When the sodium alginate and calcium solution come into contact with each other, the alginate binds with the calcium to form a gelatinous substance called calcium alginate.



pH of the liquid to be spherified plays a major factor in this process. Liquids that are too acidic might not work well for spherification (Teisha Rowland, 2020).

The ability of calcium chloride (CaCl_2) to react quickly with the alginate for the formation of divalent salt bridges and gel, makes calcium chloride the preferred source of calcium for spherification. It is known to have high solubility and can rapidly dissociate when added to a solution. But the use of Calcium chloride in reverse spherification method is limited due to the bitter taste it contributes to the final product. Therefore, in reverse spherification, where the calcium source is added in the edible liquid to be encapsulated, either calcium gluconate ($\text{C}_{12}\text{H}_{22}\text{CaO}_{14}$) or calcium lactate ($\text{C}_6\text{H}_{10}\text{CaO}_6$) can be used (P.Lee and M.A.Rogers, 2012).

III. MATERIALS AND METHODS

Sodium alginate is the main chemical used in this process, which is a polysaccharide naturally obtained from marine brown algae. Sodium alginate, Calcium chloride and Calcium lactate salts of food graded brands were purchased. Purified water and regular tap water were used for different trials. The sodium and calcium solutions prepared were transferred to closed containers and refrigerated at 4° C or 40° F. For frozen reverse spherification process, the calcium solution were frozen (at 0° C or -18° F) in ice trays or sphere molds. A calcium bath, sodium bath and clear water bath are arranged sequentially for easy working.

A. PREPARATION OF SODIUM BATH:

Sodium alginate is dissolved in water using a hand blender / mixer until it dissolves and form a milky white solution. It is then allowed to rest overnight in a refrigerator until all the air bubbles are removed. Sodium alginate concentration in water was altered as 0.5% and 1.0% w/v for different trials.



Fig 2 : Preparation of Sodium bath

B. PREPARATION OF CALCIUM SOLUTION:

6g of the desired calcium salt (Calcium chloride / lactate) is dissolved in 200mL of water (or any liquid that is to be encapsulated within the sphere) using a hand blender / mixer. This solution can be allowed to rest in a refrigerator until the air bubbles are removed, or frozen into spheres by pouring them into a mould (done for the frozen reverse spherification process). Calcium salts were dissolved in different ratios to the sodium concentration.



Fig 3 : Preparation of Calcium solution

C. REVERSE SPHERIFICATION TRIALS:

In reverse spherification, the calcium solution is dropped into the setting bath of sodium alginate. The calcium solution, sodium bath, and two clear water baths are arranged in order. The calcium solution is scooped using a round spoon and gently dropped into the sodium bath, and left undisturbed for a certain period (known as gelation period or gelling time) for the sphere formation. It is then slowly taken using a slotted spoon, and dropped into clear water baths to stop further gelation.

D. FROZEN REVERSE SPHERIFICATION TRIALS:

The calcium solution used were frozen into spheres instead of being dropped into the sodium bath as a liquid. As the sphere melts in the sodium bath, the alginate reacts with calcium and entraps liquid into a sphere. It is then slowly taken using a slotted spoon, and dropped into clear water baths to stop further gelation.

E. SOIL BURIAL TEST:

Soil burial test was done to determine the ability of the alginate spheres to degrade naturally. Spheres made using calcium chloride were buried in a small pot. The pot with small openings in the bottom to improve water and air circulation, was filled to three-fourths of its volume with moist black soil. A pit of approximately 5 cm depth was made in the center of the soil, into which the spheres were placed and covered. The pot was placed in atmospheric conditions, and the soil was kept moist with water during the entire course of testing. The weight of the spheres were weighed in regular intervals to determine weight loss, and in turn determine its rate of degradation. The test was continued until no spheres were found in the soil.

F. ONLINE SURVEY ON AWARENESS OF PLASTICS AND EDIBLE WATER BUBBLES:

An online survey was conducted via Google Forms, a survey administrative software. A survey form with multiple questions based on plastic usage, bottled water and awareness of edible packaging were framed, in order to understand consumer's awareness over SUPs, recycling of plastic bottles and acceptance of edible water bubbles in India. The survey form was shared to people of a wide age group and the total summary of responses were obtained and observed.

IV. RESULTS

The calcium and sodium solutions prepared in different concentrations were used, with varying gelation period to observe their reaction. It was observed that the gelation period had a direct effect on the diameter of the pods / spheres formed, followed by the concentration of sodium alginate.

Table 1 : Reverse spherification trials









Reverse spherification trials			
			
Trial 1	Trial 2	Trial 3	Trial 4

Table 2 : Frozen Reverse spherification trials

Frozen Reverse spherification trials			
			
Trial 1	Trial 2	Trial 3	Trial 4

A. EFFECT OF SODIUM CONCENTRATION AND GELATION PERIOD ON DIAMETER OF SPHERES:

The gelation period (immersion time of calcium source in sodium bath) was altered for different trials, in order to find its effect on the diameter of the formed spheres / pods. Diameter of the spheres were observed to vary depending on the gelation period and also the concentration of Sodium alginate concentration used. The diameter of the pods formed were measured using a vernier caliper. The pods were placed within the larger jaws in the caliper that are used to measure external diameters of objects, and the average of 3 values were calculated for each pod.

Table 3 : Average diameter of spheres

Trial no.	Alginate (% w/v)	Calcium source	Gelation period (min)	Avg. diameter of the sphere (cm)
REVERSE SPHERIFICATION				
1	0.5	Calcium lactate	10	1.45
2	0.5		5	1.40
3	1.0		5	1.53
4	1.0		10	1.60

FROZEN REVERSE SPHERIFICATION				
1	1.0 (salt water)	Calcium chloride	5	1.62
2	1.0 (tap water)		5	1.54
3	1.0	Calcium lactate	5	1.58
4	0.5		10	1.43

It was observed that the sphere made with a sodium bath prepared using salt water at 1% w/v concentration, a gelation period of 5 minutes, and using Calcium chloride had the highest diameter. The difference in calcium source, alginate concentration and the gelation period showed a considerable difference in the diameter of the spheres.

B. SOIL BURIAL TEST:

The biodegradability of the edible water bubbles were tested by the soil burial method, where the pods were buried in soil and weighed in regular intervals, to determine the weight loss, and in turn determine its ability to naturally degrade. Spheres weighing a total of 110g, made using calcium chloride were buried in a small pot with moist black soil. The weight of the membranes were weighed in a regular interval of 2 days, and degradation was observed in the rate of 9.09% initially. Rate of degradation increased as days passed, and complete degradation of the spheres were observed in 10 days.

Table 4 : Rate of degradation

Days	Weight of spheres (g)	Degradation (%)
0	110	0
2	100	9.09
4	90	18.18
6	70	36.36
8	20	81.81
10	0	100

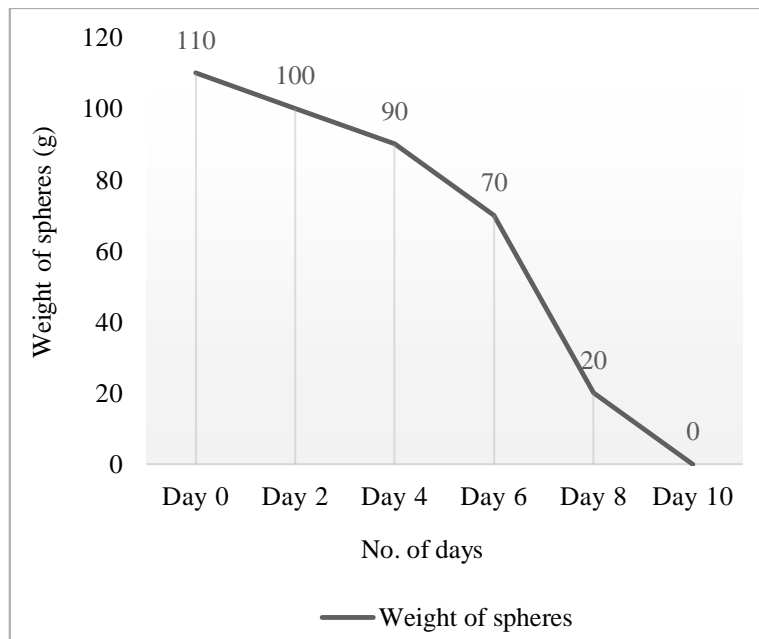


Fig. 4 : Rate of degradation chart

C. *SENSORY EVALUATION:*

Spheres made from calcium chloride and calcium lactate was given for a sensory evaluation to a group of 5 members, and the results were obtained for a total of 5, with 1 being the least and 5 being the highest score. Taste of spheres made using Calcium lactate was preferred mostly compared to those made using Calcium chloride, since it imparted a bitter taste to the spheres.

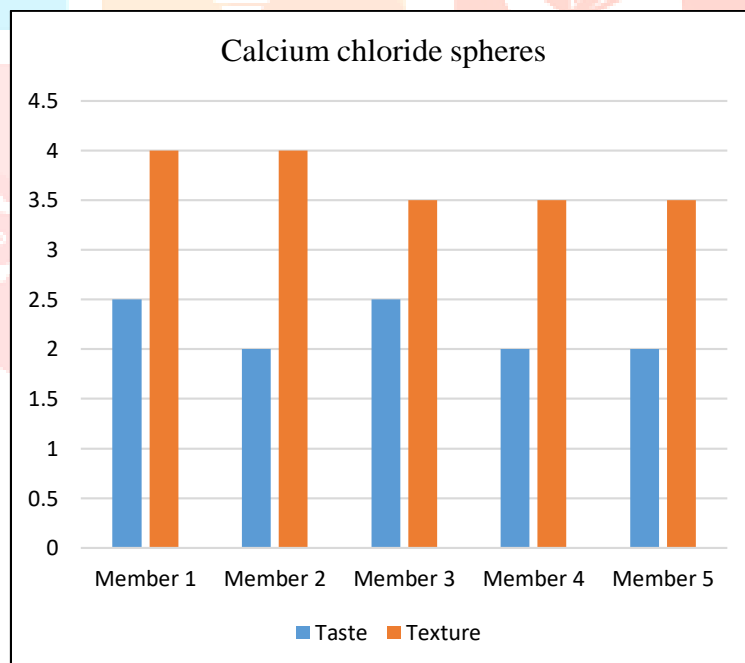


Fig. 5 : Sensory evaluation for spheres made using Calcium chloride

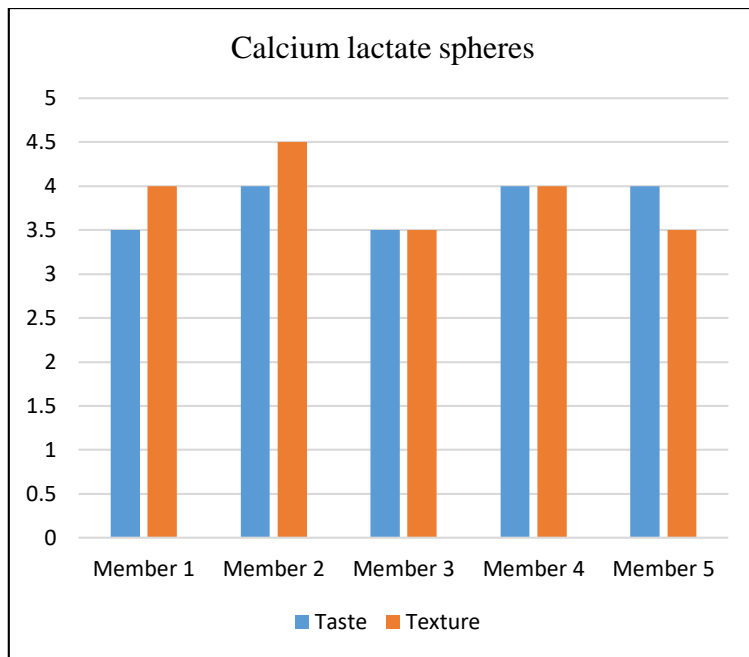


Fig. 6 : Sensory evaluation for spheres made using Calcium lactate

D. SURVEY ON EDIBLE WATER BUBBLES:

An online survey based on consumer’s awareness over SUPs, recycling of plastic bottles and acceptance of edible water bubbles, was conducted via Google Forms. A total of 230 responses were collected, and the results obtained via the survey are mentioned in the Fig. 7

This survey, thus helped in understanding Indian consumer’s thoughts on bottled water, and their current knowledge and acceptance level of edible packaging. Further research and development in this field can make edible packaging for most food products, a reality in India.

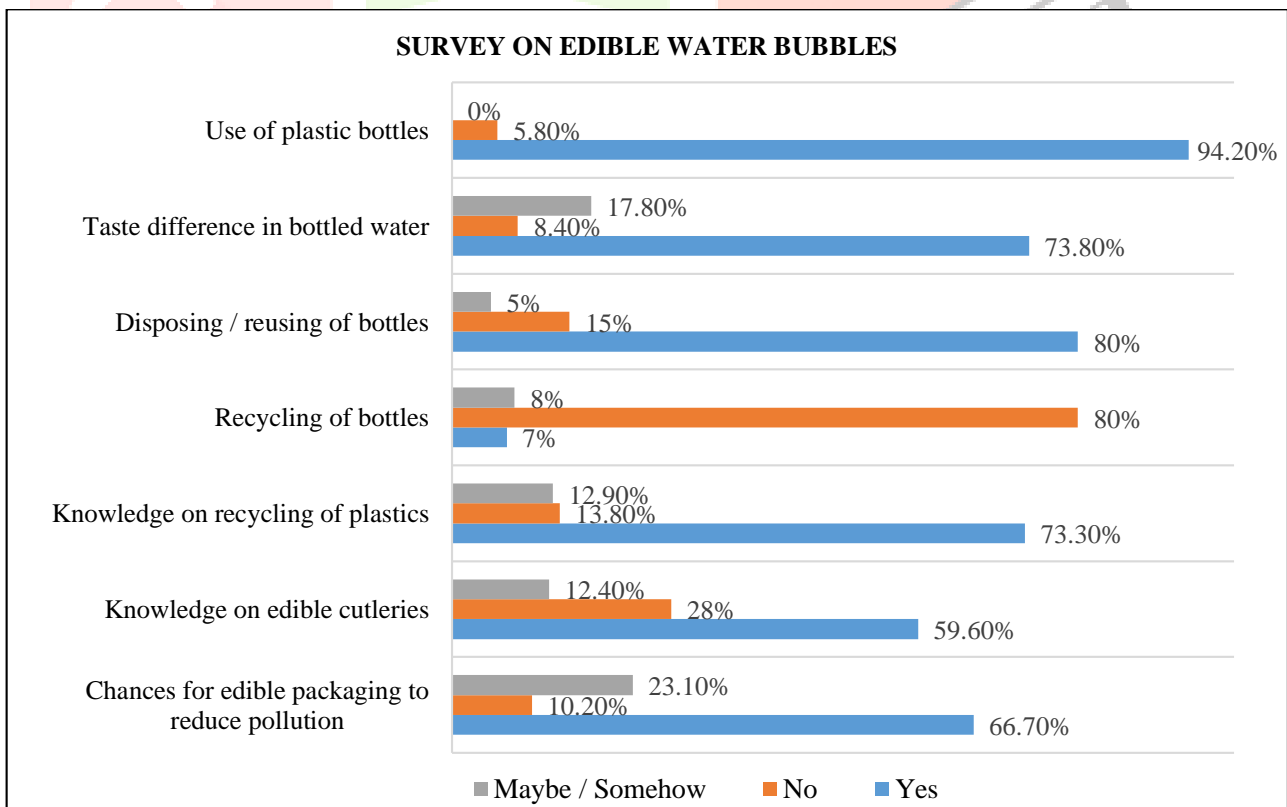


Fig. 7 : Survey results

V. CONCLUSION

The concept of edible water bubbles packaging was initiated to reduce the usage of single use plastics. Spherification, which was used mainly to produce fruit caviars or small pods of juices meant for an appealing look and immediate consumption, can now be studied further and modified in an attempt to reduce one of the most crucial issues in the modern world - plastic pollution. This type of edible packaging can be done for a wide range of liquid foods later. More research can be done on the other parameters required to enhance the physical properties of the gel membrane, thus making the spheres durable and resistant to physical stress, expand shelf life of the product etc., The survey conducted, helped in understanding Indian consumer's thoughts on bottled water, and their current knowledge and acceptance level of edible packaging. Further research and development in this field can make edible packaging for most food products, a reality in India.

VI. FUTURE PLANS

- Addition of different flavors and colors naturally into the membrane of the spheres to make it appetizing and healthy.
- Testing of detailed parameters of various factors of gel formation (required pH, temperature, drying characteristics, effect of gelling time etc.) and stability of the spheres.
- Further studies and advancements can make flavored and carbonated beverages bubbles possible, and extend their shelf life.
- Replacing single use bottles in events, by use of immediate consumption edible bubbles can be done.

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