



W/o/w multiple emulsions: A novel trend in functional ice cream preparations?

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ABSTRACT

Ice cream is a popular product worldwide. Unfortunately, it contains a significant amount of fat. In this review, promising strategies for the use of w/o/w multiple emulsion structures in creams are assessed. W/o/w multiple emulsions (MEs) enable reduction the fat without losing the creamy taste and mouthfeel and also encapsulation of sensitive compounds. The encouraging application and formation of MEs in ice cream mixtures is supported by the use of natural food ingredients, such as fiber, which helps to stabilize the whole system and improves nutritional value. The future trends may be focused on the target stabilizations using Pickering particles (PPs). The possible advantages, manufacture, evaluation methods, and predicted future prospects of MEs in ice creams are discussed.

Introduction

There is a global effort to develop new functional foods with emphasis on an enhanced nutritional value. Consumers are looking for nutritious food which also offer additional benefits and desired flavor (Erickson & Slavin, 2016). These products can be also considered as a useful source of nutrients for people suffering from some chronic or acute diseases such as diabetes (de Pinho Ferreira Guine & Joao Reis Lima, 2012), cardiovascular diseases and obesity (Papierska & Ignatowicz, 2019), inflammatory bowel diseases (Shin & Lim, 2020). Functional foods can help to manage symptoms associated with some diseases and/or prevent them. Consumption of functional foods contributes to the anti-inflammatory effects (Cohen et al., 2014), ingestion of dietary fibers reduces gastrointestinal diseases (de Pinho Ferreira Guine & Joao Reis Lima, 2012) etc. One such product is a functional ice cream prepared with w/o/w (water-in-oil-in-water) multiple emulsion (ME). Ice creams are very popular products in general and their cooling end-result after consumption has a beneficial relaxing effect on the human body (Sanagawa et al., 2020). W/o/w MEs enable a reduction of fat content and also can facilitate the encapsulation of sensitive compounds in their complex structure which protects them during digestion, especially when passing through the stomach. The targeted release of encapsulated compounds occurs in the intestine (Frank et al., 2012; Lin et al., 2020; Sun & Xia, 2020). W/o/w MEs have found applications in the field of

pharmacy (Erdal & Araman, 2006), cosmetics (Carlotti et al., 2005) and food products (Muschiolik & Dickinson, 2017). Recently published papers deal with encapsulation of microorganisms using MEs (Qin et al., 2021). The incorporation of w/o/w MEs in ice cream recipes provide extra benefits and the temperatures below zero not only avoid undesired microbial spoilage but can also improve the stability of encapsulated compounds and/or viability of probiotic microorganisms that is usually limited when these materials are added into ice cream mixtures without any encapsulation (Arslan et al., 2016). Ice cream is a complex food consisting of an unfrozen phase (serum), ice crystals, fat phase and air bubbles (Homayouni et al., 2018). Milk proteins are natural emulsifiers which have been found to work as excellent agents during w/o/w MEs preparation (Klojdová et al., 2018). In recent years there has been a trend to use Pickering particles (PPs) in emulsion stabilization (Dickinson, 2020). Pickering emulsions (PEs) differ from “conventional” emulsions in their stabilization agents. Normal emulsions are stabilized by emulsifiers whereas PEs are stabilized by a layer of solid PPs on the interface of two immiscible liquids. The properties of PPs then determine the final stability of PEs (Abdullah et al., 2020; Yang et al., 2017). Based on recent studies (Jiang et al., 2021; Jiang et al., 2021; Marefati et al., 2015; Xiao et al., 2017), it is expected to expand use towards utilization of PPs.

Abbreviations: Mes, multiple emulsions; PPs, pickering particles; PEs, pickering emulsions; w/o/w, water-in-oil-in water.

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Functional ice creams

Ice creams are very popular products with a high consumption rate in different countries. However, with regard to the health aspect, common plain ice creams suffer from high content of fat (10 – 18 %), sugar (15 – 18 %) and very low content of dietary fiber (Marshall et al., 2003a). Because of this issue, there is a tendency to meet consumer demands by preparing healthier ice creams with additional benefits. Some examples of published model preparations are summarized in Table 1.

Potential benefits of functional ice creams enhanced with w/o/w emulsions

It can be seen from Table 1 that there have been efforts to enhance ice creams with probiotics. Generally, dairy products are the main food commodity where probiotics are used as a starter culture and/or enriching ingredients. Nowadays, the second option is becoming more common because of many health-promoting properties of probiotics. However, appropriate processing of such new products remains still a big challenge because of the stability and functionality of probiotics in these (Gao et al., 2021). W/o/w MEs systems have been verified to provide the required protection of sensitive probiotics and enable their target release in colon (Qin et al., 2021). The use of MEs in ice cream preparations facilitates the protection not only of probiotics but also of different kinds of compounds, e.g. prebiotics. Prebiotics support the survival and development of probiotics and cause a favorable change of human intestinal microflora (Juárez-Trujillo et al., 2021; S. & M., 2011; Shah et al., 2020; Wilson & Whelan, 2017). An excellent source of prebiotics is fiber, which has been reported as a source of PPs (Qi et al., 2021).

Fat contents in ice creams commonly available on the market are usually between 12 and 14 % (Patel et al., 2015). An unquestionable advantage of w/o/w MEs is the partial displacement of the oil (fatty) phase by internal water phase with no influence on taste sensation. Recently, Akbari et al., 2019 have reviewed the possible ingredients which could work as fat phase replacers in ice cream. They have come to a conclusion that there is no fat replacer which would be suitable for all kind of ice cream recipes and preparations. The biggest issue were changes in sensory properties. Common used fat replacers in ice creams are maltodextrin (Karaca et al., 2009), starch (Sharma et al., 2017), dietary fibers (Soukoulis et al., 2009) and milk proteins (Mostafavi et al., 2017).

Preparation and stabilization of ice creams

Historically, the first frozen desserts were made by ice or snow flavoring. In the last century, scientists have continued to provide novel

Table 1

An overview of developed functional ice creams.

Enriching agent	Reference
Probiotics: <i>Lactobacillus acidophilus</i> La-5 (DVS), <i>Bifidobacterium bifidum</i> Bb-12 (DVS), <i>Lactobacillus reuteri</i> B-14171, <i>Lactobacillus gasseri</i> B-14168 and <i>Lactobacillus rhamnosus</i> B-445	(Salem et al., 2006)
Probiotics + prebiotics: <i>Lactobacillus rhamnosus</i> DSM 20021, <i>Lactobacillus casei</i> DSM 20011; inulin	(di Criscio et al., 2010)
Probiotics + prebiotics (goat milk): <i>Lactobacillus plantarum</i> UBLP-40; inulin	(Robins & Radha, 2020)
Amla (Indian gooseberry): source of vitamin C, phenols, dietary fiber and antioxidants	(Goraya & Bajwa, 2015)
Pomegranate by-products: source of phenolics (pomegranate peel), conjugated fatty acid - punicic acid (pomegranate seed)	(Çam et al., 2013)
Agro-industrial grape residue flour: source of phenolics, flavonoids, flavonols and anthocyanins	(Nascimento et al., 2018)
Fat replacers: Simplese (R) D-100 or inulin	(Karaca et al., 2009)
Basil oil microcapsules: antioxidant and phenolic content	(Paul et al., 2020)

ways of ice cream preparation that can deliver products meeting consumer demands (Quinzio, 2019). Ice cream processing can be realized batchwise (step by step manufacturing) or using a continuous production line.

Preparation and stabilization of regular dairy-based ice creams

Common ice creams are prepared by mixing of required ingredients, pasteurization and homogenization of the mixture, its aeration and solid. Generally, liquid phase contains ice crystals, air bubbles, milk proteins, fat globules, sugar, stabilizers and salts. The resulting textural and sensory properties are the key factors for consumer acceptability (Abbas Syed, 2018).

Preparation and stabilization of ice creams enhanced with w/o/w emulsions

Preparation processes of ice creams with w/o/w MEs require some technological modifications. The overview of functional ice cream with w/o/w ME preparation is in Fig. 1.

Preparation of w/o emulsions (fat phase)

The basic step is a preparation of an internal simple w/o emulsion (Xia & Yao, 2011) which represents the fat phase of the final functional ice cream. In this step, the internal water phase, which may contain sensitive compounds, is mixed with oil (fat) phase with a lipophilic emulsifier or relevant PPs to create a stable w/o emulsion. This w/o emulsion can be then added to the other components before undergoing further processing. For the subsequent processing, it is necessary to avoid any instability of the w/o emulsions, namely flocculation, coalescence and Ostwald ripening (Iyer, 2008). So, the most crucial step is the choice of oil (fat) phase, an appropriate lipophilic emulsifier and/or PPs for interfacial stabilization. With respect to the intended preparation

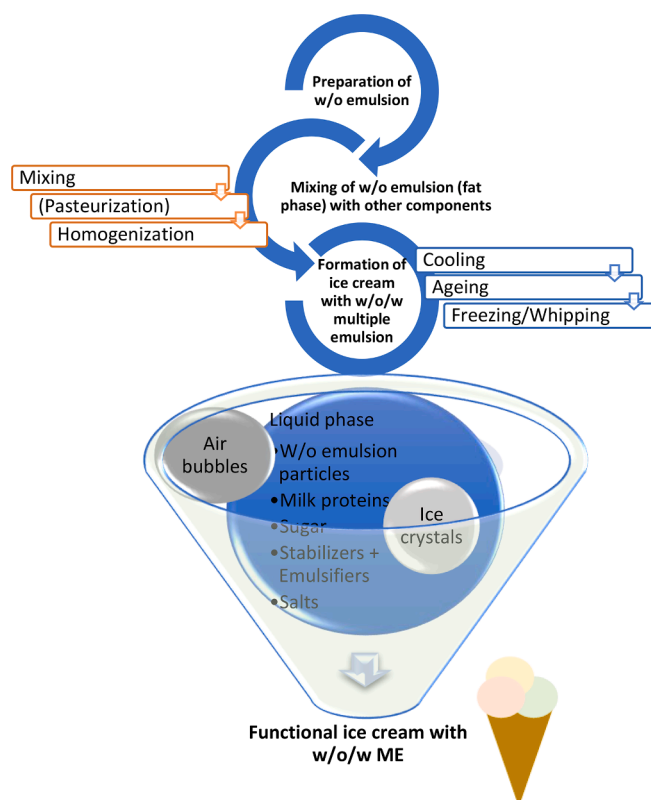


Fig. 1. Preparation overview of functional ice creams with w/o/w MEs.

of healthier ice creams, a stabilization with natural agents is a big challenge. For the stable internal w/o emulsions, PGPR (E 476—polyglycerol polyricinoleate) has been commonly used. PGPR is a mixture of products formed by the esterification of polyglycerols with condensed castor oil fatty acids; has “food safe status” but its levels in foods are regulated (Mortensen et al., 2017). The most natural stabilization may be provided by food-grade PPs. This topic has been studied very intensively in recent years (Chen et al., 2020; Linke & Drusch, 2018; Murray, 2019; Niroula et al., 2021), also with respect to an effective use of by-products from food industry (Gould et al., 2016; Sarkar & Dickinson, 2020) to fulfill current requirements on sustainability (Lau et al., 2021). Additionally, PPs can be a valuable source of nutrients, e.g. fiber (Qi et al., 2021). PPs create single-layered or multi-layered film on the interface and the resulting stability of the emulsion is determined by the particles' composition and properties (type, shape, size, etc.) (Yang et al., 2017). The preparation process of w/o emulsions can be conducted using a variety of technical equipment on a conventional industrial scale or on a laboratory experimental scale: homogenizers, high pressure homogenizers (Mohan & Narsimhan, 1997), ultrasonic homogenizers (Jiang et al., 2021, Jiang et al., 2021), membrane (Jing et al., 2006) and microfluidic techniques (Chu et al., 2007).

Preparation of ice cream mixtures and their processing

At this stage of production, all ingredients (liquid and solid phases) are usually mixed with a blender. Taking into account the nature of chosen ingredients, some pre-heating (around 40 °C) can be provided to improve homogeneity (Mostafavi et al., 2017). During mixing, the proper structure of w/o/w ME is formed because the simple w/o emulsion is mixed with the other ingredients and liquid surrounding phase represents the external water phase of w/o/w ME. Many of the ingredients help to stabilize w/o/w MEs naturally. Milk proteins are natural emulsifiers as a lecithin and an addition of milk powders or concentrates improve the stability of w/o/w emulsion (Shokri et al., 2022; Sivapratha & Sarkar, 2018), (Alhaji et al., 2020; Deng, 2021). Gums, hydrocolloids in general (Abbasi, 2017; Dickinson, 2003) and fiber help to avoid the aggregation of the w/o particles and some of them may work as PPs for the interface between oil phase and external water phase in the created w/o/w ME (Cui et al., 2021). Artificial emulsifiers, e.g. Tweens, when added, provide an additional stabilization of the whole emulsion systems (Ma & Chatterton, 2021; Mohd Isa et al., 2021). Consequently, the step of pasteurization is required to ensure a microbiological safety (Cook & Hartel, 2010). Potentially, this is a critical step for MEs because their complex structure can be disrupted by the action of heat. (Bou et al., 2014) described a decreased stability of w/o/w MEs after heat treatment (70 °C, 30 min). On the other hand, we have found in our previous work (Klojdová et al., 2022) that model manufacturing of w/o/w MEs (5 to 85 °C, shear rate 0.1 to 1000 s⁻¹) did not significantly influence their encapsulation efficiency of glucose. An alternative way for the step of some heat treatment may be an aseptic preparation process with pre-treated ingredients. This design of the preparation is much more difficult to implement (Reuter, 1998) and a homogenization, the next step in ice cream manufacture requires higher temperature anyway. For functional ice creams with w/o/w MEs the homogenization is a beneficial step because it causes the decreasing of fat particles (w/o emulsion droplets) under 1 µm. This decrease improves the stability of w/o/w MEs and a high pressure homogenization is commonly used step during MEs preparation (García et al., 2016). To obtain an ice cream mixture with required properties, such as smooth texture, the mixture is passed twice through a high pressure valve homogenizer. The higher the fat and total solids in the mixture, the lower the pressure should be. Commonly, pressures ranging between 6 and 20 MPa are used (Biasutti et al., 2013; Hayes et al., 2003).

After finishing mixing, the homogenized smooth mixture for ice cream preparation is cooled to 5 °C and its ageing should be provided. During ageing of ice cream mixture, the proper hydration of milk

proteins and other stabilization agents takes place, as well as the crystallization of fat globules (Dogan & Kayacier, 2007). In functional mixtures with w/o/w MEs, w/o droplets (globules) must be stabilized enough to avoid their disruption during the crystallization. However, the crystallization of oil phase in w/o droplets can support the stabilization of these systems due to the creation of a solid barrier made from fat layer (G. Li et al., 2021; Relkin et al., 2003). Fat crystals can be also considered as agents for Pickering stabilization of emulsions (Ghosh & Rousseau, 2011; Rousseau, 2013). Temperatures for ageing can be different with regard to required properties of final ice creams (hardness etc.) (Akalın et al., 2008). While Goff (Goff, 1997) used an ageing setting of the mixture at 2 to 4 °C for 2 to 4 h, more recent Dogan & Kayacier (Dogan & Kayacier, 2007) have demonstrated advantage of longer ageing period of 24 h at 0 °C for higher viscosity and smooth texture.

One of the important steps in ice cream manufacture is freezing. Aged ice cream mixture is frozen quickly while being stirred and whipped at temperatures around -5°C to incorporate air bubbles and limit the grow of ice crystals (the size should be less than 30 µm) (Marshall et al., 2003b). The final shape and size of ice crystals influence the smooth and creamy mouthfeel (Goff, 2008). For manufacturing, scraped-surface freezers are usually used (Kowalczyk et al., 2021). Recently, the application of ultrasound for formation of small ice crystals and better stability of fat globules during freezing and crystallization steps has been demonstrated (Akdeniz & Akalin, 2019). For functional ice creams with w/o/w MEs the crucial step is to ensure the emulsion stability during freezing and avoid the damage of emulsion systems by ice crystals (Aronson et al., 1994). Generally, in w/o/w MEs, the fat layer which surrounds the internal water phase must have a stabilizing effect (Ghosh & Rousseau, 2011).

After the freezing step, the final ice cream products are frozen at storage temperature below -10 °C (Aritonang et al., 2019). The higher the storage temperature of ice creams the bigger ice crystals can occur, especially on the surface of the product, when the temperatures are higher as -5°C (Donhowe & Hartel, 1996). This behavior may also cause the damage of w/o/w ME structures because of any potential influence of ice crystals on w/o globules integrity. For functional ice creams with w/o/w MEs content, it is also important to consider the physico-chemical properties of the complex system. The storage temperature affects the state of aggregation of internal water phase which is entrapped in fat particles. Additionally, the structure of w/o/w MEs must be stable during partial melting before consumption (Muse & Hartel, 2004).

Evaluation methods of functional ice creams enhanced with w/o/w multiple emulsions

(Tekin et al., 2017) have published analysis of a model ice cream with a ME which included overrun, meltdown rate and rheological measurements. These measurements are very important for a complex evaluation of general properties of ice cream and probably enough for ice creams where MEs are used to replace fat only. Due to the complexity of ice creams with w/o/w MEs where any functional components are encapsulated in internal water phase, additional methods for their evaluation are required (Saffarionpour & Diosady, 2021).

Sensory properties

For consumers, the sensory quality of ice creams is the most important aspect. The behavior of ice cream during the consumption is governed by the quality, quantity and size of components in ice cream mixture (Amador et al., 2017). For ice creams with MEs, the main sensory aspect is to avoid changes in mouthfeel compared to consumption of conventional ice cream (Oppermann et al., 2016) similar to replacement of sugar by oligosaccharides (S. Yang & Choi, 2007). Generally, the overall sensory acceptability for ice creams with and within MEs have been evaluated to be similar. The changes in sensory properties have

been observed when e.g. guar gum or gum tragacanth were used for stabilization (Tekin et al., 2017). Changes in taste during consumption of ice creams enhanced with MEs can be caused by thermodynamic instability: internal water phase leakage (Khadem & Sheibat-Othman, 2019) (e.g. bitter encapsulated compounds (Edris & Bergnsthäl, 2001), changes in particle sizes of ingredients (Khadem & Sheibat-Othman, 2020) (especially fat globules (Leister et al., 2022)), off-flavor of emulsifiers and stabilizers (L. Li et al., 2015) etc. To prevent any undesirable mouthfeel during the consumption of ice creams with MEs, attention must be paid to proper manufacture and stabilization. Here, the use of PPs can be very beneficial in the future (Zhu et al., 2018).

Physico-chemical properties

In this section, the most important methods and properties for the objective quality assessment of ice creams with MEs are discussed.

Texture

The texture of formulated ice creams with MEs strongly depends on the matrix consisting of air bubbles, w/o emulsions (fat globules), ice particles and an unfrozen aqueous solution (Goff, 1997). For ice creams with w/o/w structures, key requirements for a proper texture are stable w/o emulsions, which determine the smooth full cream consistency (Fuangpaiboon & Kijroongrojana, 2017) and a desirable action of stabilizers (Baer et al., 1997). Stabilizers and emulsifiers can negatively affect the texture because of their tendency to be gritty (milk proteins – formations of large protein aggregates) (Morell et al., 2017). They also influence the thickness of the unfrozen phase and the creation of ice crystals (Miller-Livney & Hartel, 1997).

Melting rate and overrun

Melting rate is a complex process which affects the structure of ice creams with w/o/w MEs significantly. Generally, the size and distribution of air bubbles affect the final hardness and overrun of ice creams (Marshall et al., 2003a). The heat transfer is governed by ingredients in the mixture, where air bubbles are the most important insulator. Thus, ice creams with high overrun have a tendency to melt down slowly (Sofjan & Hartel, 2004). However, the high overrun can cause a destabilization of fat globules (Warren & Hartel, 2018). This issue must be particularly considered for mixtures with MEs because the stability of fat globules (w/o particles) is required (Neumann et al., 2018). Also during melting and consumption, it is necessary to ensure the proper integrity of w/o particles. Ice crystals are a good heat conductor and their size and concentration in w/o/w matrix affect melting rate and stability of w/o particles. The temperature changes can help to disrupt the emulsion structure. The promising stabilization could be provided by solid particles, i.e. by the PPs. The thermodynamic stability of PEs is improved (Kumar et al., 2021) and may offer additional dietary benefits (Qi et al., 2021).

Rheological properties

Rheology of prepared ice creams with w/o/w MEs provides a general quality control of products. The rheological properties of final products offer an overview on complex behavior during storage and consumption (Freire et al., 2020). Rheological behavior of ice creams is mostly influenced by ice crystals (Granger et al., 2005). However, partly coalesced fat globules can also cause changes in rheological properties of the matrix (Goff & Hartel, 2013). To avoid coalescence of w/o particles, the stabilization properties of the whole matrix are crucial. When w/o particles undergo a coalescence the result rheological properties are completely different because of bigger particles and changes in water–oil proportion of the w/o emulsion (Khadem & Sheibat-Othman, 2017). Moreover, leakage of internal water phase occurs more easily (Schuch et al., 2014). The information on rheological properties of ice creams with MEs can be obtained by a combination of oscillatory, transient and large deformation measurements (Freire et al., 2020).

Particle size and microscopic observation

While rheology is a tool for general quality assessment of ice creams, a particle size measurement provides specific integrity evaluation of ice creams with w/o/w MEs. Almost all instability behavior of w/o/w systems (coalescence, Ostwald ripening etc.) is accompanied by changes in the size of fat particles. Thus, microscopic observation of the matrix can highlight the potential instability process. Advanced techniques, such as fluorescent microscopy enable the observation of leakage mechanisms occurred in w/o/w systems (el Kadri et al., 2018). Other useful tools for particle size measurements are laser diffraction (Kori et al., 2021) and dynamic light scattering (Oluwole et al., 2020).

Encapsulation efficiency of w/o/w emulsions

The evaluation of encapsulation efficiency of ice creams enhanced with w/o/w structures is the important measurement especially for ice creams with encapsulated sensitive compounds (with w/o/w structures not only to reduce fat content) (Heidari et al., 2022). The encapsulated amounts of bioactive compounds or even microorganisms may be monitored by microscopic observations, ELISA techniques (Paula et al., 2018), target color (Sebben et al., 2022), evaluation of the number of microorganisms (Qin et al., 2021) etc.

Conclusions and future prospects

The use of w/o/w MEs in ice creams is highly advantageous. The incorporation of these complex structures enables the reduction of fat content in the final product, without any significant changes in mouthfeel, and the encapsulation of sensitive compounds in the internal water phase. Ice cream is a very popular product on the market and its “healthier modification” represents a functional food. Moreover, the ice creams mixture is a rich source of emulsifiers and stabilizers anyway. Milk proteins stabilize the internal w/o emulsion and together with other components e.g. fibers from different sources of fruits or vegetables can govern like a PPs. Another stabilization effect may be provided by fat crystals. Given the complex structures of MEs systems and growing consumer demands for functional products with added value, ice creams with MEs can be interesting items on the market. The use of natural ingredients for stabilizations during manufacture enhances nutritional value. In the future, we expect use of new food materials, often by-products, for the stabilization of systems with MEs. This assumption is in line with the current trend of expanding investigation of PEs prepared with natural PPs. Based on these findings from recent years, we expect w/o/w MEs will play a major in functional ice cream preparations.

CRediT authorship contribution statement

Iveta Klojdová: Conceptualization, Writing – original draft. **Constantinos Stathopoulos:** Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

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