Chapter 4
Modernization of the Traditional Irish Cream Liqueur Production Process

Peter C. Mitchell

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4.1 Introduction

The spirit drinks sector in Ireland has a long tradition and strong links to the agricultural sector. Whilst whiskey is the spirit drinks category with the greatest heritage in Ireland, it is the cream-based liqueur category which has grown to prominence in Irish food history. Today, Baileys as the number one selling liqueur brand in the world leads the 126 million litre global cream-based liqueur category, with a 46 %

P.C. Mitchell (✉)
Northern Ireland Centre for Food and Health, University of Ulster,
Coleraine, Northern Ireland, UK
e-mail: pc.mitchell@ulster.ac.uk

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volume share (Cunnington 2010). The rest of the category is made up of a number of small global brands and small regional/local producers (Cunnington 2010).

Varnam and Sutherland (1994a) state that liqueurs may be defined as distilled spirits, which have been sweetened and flavoured with substances of compatible taste. Traditional methods and practices prevailed in the manufacture of many liqueurs, until their classification became better defined, particularly in Europe and the USA. As a sub-classification of spirit drinks, a liqueur must contain at least 15 % alcohol by volume (Clutton 2003). However, there are significant differences in the liqueur definitions within Regulation (EC) No 110/2008 of the European Parliament (European Commission 2008) and the Federal Alcohol Administration Act (Alcohol and Tobacco Tax and Trade Bureau 2007), particularly in terms of minimum sugar content. However, the definitions still allow for a wide variety of liqueurs to be produced.

Commercial cream liqueurs are added value, long-life, oil-in-water emulsions, combining the flavour of an alcoholic drink with the texture of thickened cream. To control the kinetics of the processes that lead to the breakdown of cream liqueurs requires a device to break up the milk fat globules and disperse them within the emulsion system, and the addition of stabilizing chemical additives (Bergenstahl 1995) to reduce the interfacial tension and prevent flocculation and coalescence of the dispersed phase (Kinsella and Whitehead 1989). The dispersed phase is more likely than not to be partially solidified, and to an extent which is quite strongly temperature dependent, and thus reference is made interchangeably within this review to dispersed phase droplets and particles. The high-pressure homogenizer and caseinates are the respective widely used device and stabilizer in the production of traditional Irish Cream liqueurs. The inclusion of caseinate means that the product cannot be consumed with a mixer drink, as the resulting pH drop would cause protein precipitation. Traditional Irish Cream liqueurs are some of the finest food emulsions (Dickinson and Stainsby 1988) where an exemplar manufacturing specification is 90 % of particles less than 0.5 μm. The particle size of the dispersed phase droplets influences not only storage stability but also the palatability, mouthfeel, texture and general appearance of traditional Irish Cream liqueurs.

Whilst the technology platform for the manufacture of stable cream liqueurs has been in place since 1980s (Banks and Muir 1988), the commercial importance of these products continued to drive scientific and technical investigations aimed at increasing volume of production and range of products, and enhancing product quality in a more demanding global supply chain. Today the use of alternative sources (Medina Torres, Calderas, Gallegos-Infante, González-Laredo, and Rocha-Guzmán 2009) and new types of ingredients, and enhanced process technologies (Heffernan, Kelly, and Mulvihill 2009) are under investigation in order to lower the cost of manufacture and formulate novel products. So in just over 35 years, traditional Irish Cream liqueur products and production processes have evolved and modernized. This literature review will focus on the problems encountered and solved, whilst emphasizing the control of homogenization parameters, in the production of stable, high-quality traditional cream-based liqueurs.

Table 4.1 Composition of a traditional cream liqueur

Component	Traditional product example
Milk fat	16 % w/w
Added caseinate	3.3 % w/w
Added sugar	19 % w/w
Ethanol	14 % w/w[a]
Total solids	40 % w/w
Fat to protein	4.2

[a]Corresponds to 17 % v/v

4.2 Irish Cream Liqueurs

The composition of cream liqueurs can vary widely with milk fat, added caseinate, added sugar and ethanol content ranging from 4.5 to 16.5 % w/w, 1.8 to 3.8 % w/w, 17.5 to 22 % w/w and 12.7 to 15.4 % w/w, respectively (Banks et al. 1981a). The traditional Irish Cream liqueur is a premium product, containing whiskey as part of the spirit content and a composition which is at high end of the milk fat range. The composition of one commercial traditional Irish Cream liqueur is shown in Table 4.1.

The manufacture of cream liqueurs is detailed by Banks et al. (1981a), and Banks and Muir (1985, 1988). The two methods of manufacture discussed are the single-stage and two-stage methods. In the former, caseinate and sugar are dissolved in the water at 85 °C by vigorous stirring, and the air is dispersed from the mixture. Next double cream is blended into the mixture, and alcohol is added. This pre-emulsion is then heated to 55 °C and homogenized at 27.6 MPa (4000 psi). The two-stage method differs in that alcohol is added after homogenization. An example of a commercial single-stage process flow diagram for the production of traditional Irish Cream liqueurs is shown in Fig. 4.1. This highlights the importance of homogenization parameters, including the use of the two-stage valve and multiple passes, which will be covered in Sect. 4.3.

The formulation, testing and stability of 16 % fat by weight cream liqueurs have been researched (Power 1996). Cream, ethanol stability, the defects associated with cream liqueurs and the key controls to prevent and minimize destabilizing mechanisms merit particular attention.

4.2.1 Cream

Cream is produced from unhomogenized milk by mechanical separation of the fat globules. Cream is an oil-in-water emulsion where the globules are dispersed in an aqueous (serum) phase consisting of protein, lactose and salts. Cream is defined in the UK as “that part of milk rich in fat that has been separated by skimming or otherwise”, and legally contains not less than 12 % fat by weight (Early 1998).

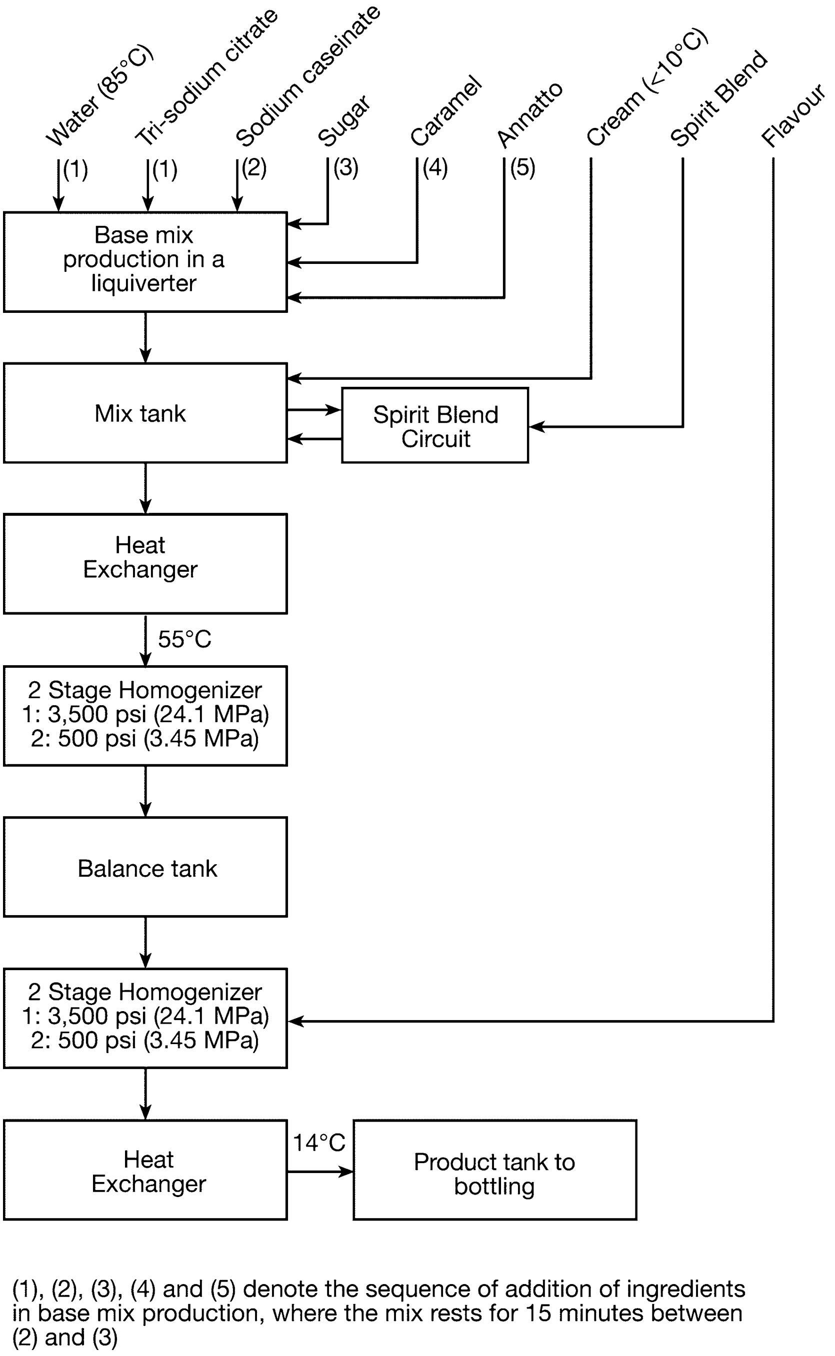


Fig. 4.1 Example of a commercial single-stage process flow diagram for the production of traditional Irish Cream liqueurs

The World Health Organization standards for the minimum fat content by weight of creams are: half, 10–18 %; single, 18 %; whipping, 28 %; heavy whipping, 35 % and double, 45 % (Varnam and Sutherland 1994b).

Cream suitability for processing relates to fat content, acidity, seasonality, storage, handling and heat treatment (Rothwell 1989; Towler 1994). Cream homogenization and increased storage time increase cream viscosity. Winter cream has poorer ethanol stability in comparison to cream separated from mid-lactation milk (Rothwell 1989).

Double cream is the most usual source of milk fat in traditional Irish Cream liqueurs. Double cream contains surface active materials at *circa* 0.55 % by weight, which includes protein and the naturally occurring milk fat globule membrane (Anderson 1991). The protein to fat ratio in double cream is *circa* 0.04 (Muir and Banks 1986). When double cream undergoes slight homogenization, fat globules clump together and cluster, and there is a marked thickening of the cream (Mulder and Walstra 1974). Cream liqueurs employ a much higher protein to fat ratio, and also use more severe homogenization pressures (Muir and Banks 1986). It is the use of double cream at *circa* 33 % of the formulation that gives the traditional Irish Cream liqueur its richness and body, and masks the harshness of the alcohol (Muir and Banks 1984).

4.2.2 Ethanol Stability

The addition of ethanol is used as an indicator of milk stability. The higher the calcium concentration, the less stable the caseinate complex to ethanol (Davies and White 1958). Horne and Parker (1980) increased the ethanol stability of milk by increasing the pH above 7. Horne and Muir (1990) demonstrated that the level of free calcium controls the ethanol stability of the milk system. This finding has proved extremely useful in the production of cream liqueurs. The inclusion of citrate to sequester calcium prevents calcium-associated instability eliminating one of the major defects of cream liqueurs (Banks et al. 1981a).

4.2.3 Defects Associated with Cream Liqueurs

The main defects which occur in cream liqueurs are: (1) creaming, where the fat forms a layer at the surface of the liqueur; (2) formation of a plug of cream or fat in the neck of the bottle; (3) gelation of the bulk of the product with some accompanying syneresis and (4) appearance of a slight granular precipitate at the bottom of the bottle (Banks and Muir 1985; Banks and Muir 1988; and Dickinson, Narhan, and Stainsby 1989).

Creaming is the gravitational separation of emulsified droplets. This results in the formation of layers, with oil volume fractions higher than that of the original emulsion. The creamed layer is usually visible to the naked eye.

Creaming in cream liqueurs is usually reversible as gentle shaking will redisperse the fat layer. The formation of a cream plug or "cohesive cream" in cream liqueur is more likely in cream liqueur formulations at low pH, high calcium content or low emulsifier content. The effect of these factors will be increased substantially by temperature fluctuations during storage (Banks et al. 1981a; Dickinson et al. 1989).

The type of gel which may form due to instability in cream liqueurs is classified as a particle gel (Clark 1992; Power 1996). This gel undergoes syneresis with the separation of a clear liquid at the bottom of the bottle.

The sodium or potassium salts of citric acid may be used to sequester calcium in emulsions. Ionic calcium has a major effect on the stability of dairy products including cream liqueurs (Banks et al. 1981a; Davies and White 1958; Horne and Muir 1990). Citrate binds with the calcium, preventing instability. The appearance of a slightly granular precipitate has occasionally been observed at the bottom of the bottle. This is believed to be excess citrate. Citrate complexes the calcium in the liqueur and reduces the possibility of gelation and thus increases the shelf life at ambient temperature from months to years (Banks and Muir 1988). In the traditional product example shown in Table 4.1, tri-sodium citrate di-hydrate is used at a level of 0.19 % by weight of the total formulation.

4.2.4 Controls to Prevent and Minimize Destabilization of Cream Liqueurs

Steric (polymeric) repulsion, electrical double layers (electrostatic repulsion) and increased continuous phase viscosity are the main mechanisms for the stabilization of food emulsions (Schubert and Armbruster 1989). Steric repulsion, attributed to the protruding part of the casein molecule, and the repulsion by charged protrusions of the particles with similar charges are very important in the formation of stable cream liqueurs (Narhan 1987). As the alcohol level increases in a cream liqueur, there is a reduction in steric repulsion and an increase in the viscosity of the continuous phase, with a resultant increase in particle aggregation and coalescence (Banks and Muir 1985). However, alcohol-induced aggregation will occur above 17.5 % w/w alcohol strength, which must be avoided (Banks and Muir 1988). Excess heat treatment of cream must also be avoided in the production of traditional Irish Cream liqueurs so as to prevent aggregation by cross-linking (Power 1996). The addition of tri-sodium citrate to bind ionic calcium, which would otherwise aggregate casein micelles, and ensuring the pH is above 6.8 are also important controls in the production of traditional Irish Cream liqueurs (Muir 1989). Temperature controls are also important so as to minimize fat crystallization, which could result in bridging between particles, and to avoid temperature cycling with agitation, which could result in the desorption of the protective protein layer (Narhan 1987).

4.3 Homogenization Parameters

In cream liqueur production, the term "homogenization" and "homogenizer" refer to the process and equipment related to the classically recognized homogenizer, which was first developed by Auguste Gaulin for milk treatment (Tunick 2009). In such a high-pressure homogenizer, a positive displacement pump, usually of plunger or piston type, is used to force the liquid into the homogenizing valve where mechanical energy is used to break drops into smaller droplets (Wilbey 1992).

The premix enters the valve seat at a relatively constant rate of flow, relatively low velocity and high pressure (10–40 MPa or 1450–5801 psi) and, as it begins to move into the narrow adjustable gap between the valve and the seat (15–300 μm), it undergoes a very rapid rise in velocity and decrease in pressure causing turbulence, cavitation and intense mixing. For example, a pressure drop of 13.8 MPa or 2000 psi causes a velocity in excess of 160 m/s and the whole process of homogenization is complete in less than 50 μs (Pandolfe and Kinney 1983). Turbulence on the low-pressure side of the valve is probably the most important factor leading to the formation of fine droplets (Dickinson 1992). Whilst the correlations between characteristics of turbulence and cavitation, parameters of homogenizing valves and homogenization efficiency are insufficiently known (Rovinsky 1994), much is now understood about the efficient operation of high-pressure homogenization in Irish Cream liqueur production. Current understanding of homogenization parameters is now briefly discussed under the following headings: formulation; premix quality; valve technology; homogenization process; and troubleshooting protocol.

4.3.1 Formulation

Important characteristics of formulation which influence homogenization efficiency are the amount and type of emulsifier, the dispersed phase concentration and the viscosity of the dispersed and continuous phases.

The emulsifier lowers the interfacial tension between the two phases being homogenized, and therefore makes more efficient use of the available energy. Less homogenizing energy is required to overcome interfacial tension. Also, the emulsifier stabilizes the new interfacial area formed during homogenization and later prevents coalescence and agglomeration of the droplets. The cost of emulsifier is many times more than the cost of the mechanical energy thus the high-energy homogenizer is more economical than the low-energy mixer for emulsification (APV n.d.c). If the concentration of the emulsifier is insufficient or the wrong type of emulsifier is used, overworking occurs (Pandolfe 1995). This can be recognized if an increased average droplet size or even the formation of two separate phases of the emulsion is obtained on increasing the homogenization pressure. This is because the emulsifier cannot cover the expanded surface of the smaller droplets leading to a rate of coalescence which exceeds the rate of disruption. At a low protein to fat ratio of 0.10, Muir and Banks (1986) found that the long-term stability of a traditional cream liqueur, which used sodium caseinate as the stabilizer, was poor, and when such a premix was subject to severe homogenization, it could lead to the formation of large particles with subsequent creaming and fat plug formation. Whilst the protein to fat ratio is 0.21 in the traditional Irish Cream liqueur example (Table 4.1), there are other stable 16 % w/w fat and 17 %v/v traditional Irish Cream liqueurs which use a lower protein to fat ratio by combining a low molecular weight emulsifier, such as the nonionic glycerol monostearate (Euston 2008), with a reduced level of sodium caseinate in the premix. In the production of cream liqueurs there are advantages to be gained from using certain types of caseinate (Muir and Dalgleish 1987), and thus manufacturers have a preferred caseinate supplier. Lynch and Mulvihill (1997) concluded that changes in the apparent viscosity of cream liqueurs on storage at 45 °C are caseinate dependent, and suggested that electrostatic and sulphydryl interactions may be involved in these changes. Medina Torres et al. (2009) also observed significant changes, with storage time at 40 °C, in apparent viscosity and particle size distribution between caseinate batches which differed in metallic ion content. The best caseinate batch for longer term cream liqueur stability and reduced age thickening had the lowest total metallic (Ca^{++} and Na^{+}) ion content.

As the concentration of the dispersed phase is increased, the probability of the droplets not being reduced in size is increased (Pandolfe 1995). At a constant fat to emulsifier ratio and constant homogenization pressure, the increase in average particle size with increased dispersed phase concentration from 4.5 to 16.5 % w/w milk fat would be very small. However, once above a minimum milk fat volume fraction which is homogenization pressure dependent, the average particle size will increase with increasing dispersed phase concentration (Phipps 1983). When the emulsifier is required to handle an increasing surface area, it may not be able to stabilize the interface before droplets collide and coalescence occurs.

As the dispersed phase viscosity is increased, the average particle size of the homogenized product decreases. As the continuous phase viscosity is increased, there is a slight increase in the average particle size of the homogenized product, but above a viscosity of 50 mPa.s (cP), the average particle size remains constant (Pandolfe and Kinney 1983). The specification for apparent viscosity at 25 °C and 39.6 s^{-1} shear rate for the traditional Irish Cream liqueur example given in Table 4.1 is 28–38 mPa.s (cP).

4.3.2 Premix Quality

At any given homogenizing pressure, a fixed amount of energy is available for particle size reduction. If a significant portion of this energy is needed to reduce very large particles, then there will not be enough energy left over to work on the smaller particles and reduce them even further (Masucci 1989a). Thus to maximize homogenization efficiency, it is important to improve premix quality, as measured by uniformity and smaller particle size, whether by altering raw material quality, the formulation, the batch preparation method or the mechanical premix device employed (Masucci 1989a; Pandolfe and Kinney 1983). In cream liqueur production, the premixing of phases to form a pre-emulsion must be preceded by the dispersion of the powdered ingredients. Caseinates are very cohesive and have a tendency to form agglomerates. Caseinates are difficult to dissolve and will rapidly increase in apparent viscosity, especially if added directly to cream (Silverson n.d.). In cream liqueur production, long mixing times, the resting of caseinate after dissolution and the testing of the pre-emulsion prior to homogenization for % of particles less than 2 μm and apparent viscosity (39.6 s^{-1} shear rate) at the temperature at which homogenization occurs (55 °C), have traditionally been used to ensure an acceptable premix quality. In recent times, the addition of a high-shear in-line mixer to the existing cream liqueur production process has been adopted by one manufacturer so as to give agglomeration-free dispersion of caseinate, rapid mixing times, a more uniform, stable pre-emulsion of low particle size and faster processing through the high-pressure homogenizer (Silverson n.d.). Short- and long-term stability benefits have been realized in cream liqueur production practice, although use of the in-line mixer can lead to greater age thickening of the cream liqueur.

Inclusion of air in the raw product, during premixing but more frequently by leakage through the seals and/or gaskets of the homogenizer system, reduces homogenization efficiency and results in the formation of a cream line shortly after homogenization as very small fat particles which would not have separated otherwise adhere to the surfaces of the air bubbles which escape to the surface of the product. Increasing the homogenizing pressure will result in even smaller bubbles of air; therefore, an even more severe separation of the fat occurs as these air bubbles escape to the surface (APV, n.d.a).

4.3.3 Valve Technology

The key to any homogenizer is valve technology. Whilst there a wide variety of high-technology valves for different applications (Pandolfe 1999), a standard "plug-flow" valve geometry configured as a two-stage valve assembly is used for most emulsions, including cream liqueurs. An exception in dairy emulsions is the use of a Gaulin Micro-Gap homogenizing valve assembly in high volume liquid milk processing to produce the same or better product quality at up to 40 % lower pressure, and thus reduced energy cost, and with increased valve life (Pandolfe 1999).

In a two-stage design, the second stage establishes controlled backpressure, concentrating the energy in the first homogenizing zone and also minimizing the possibility of clumping/coalescence. In evaluating valve combinations, it has been found that with fluid milk at any given homogenizing pressure, efficiency is increased by the use of a second-stage valve, whereby 10 % to a maximum of 20 % of the total pressure is applied by the second-stage valve (APV, n.d.b). To give the desired total pressure, which can be 27.6 MPa (4000 psi) in cream liqueur production, the second-stage valve is set first, at for example 12.5 % of the total pressure (3.45 MPa; 500 psi), followed by the first-stage valve, to give an actual pressure drop of 24.1 MPa (3500 psi) through the first-stage valve.

4.3.4 Homogenization Process

In the homogenizing valve, according to turbulence theory it has been shown that the homogenizing pressure (P) is related to the average volume/surface diameter (d_{vs}) by Eq. (4.1)

$$d_{vs} \text{is proportional to } P^{-0.6} \tag{4.1}$$

So increasing the homogenizing pressure will reduce the average particle size and increase the energy input costs (Pandolfe and Kinney 1983). Banks et al. (1981b) reported the typical total pressure for homogenization in pilot plant cream liqueur production as 27.6 MPa (4000 psi), but with a requirement for a second pass through the homogenizer at the same total pressure. Muir and Banks (1986) conducted their pilot scale work on multiple homogenizations of cream liqueurs at 31.0 MPa (4500 psi). Widmar and Tripp (1990) presented their single pass process, using a total pressure for homogenization of 34.5 MPa (5000 psi), for the preparation of cream-based liqueurs.

The temperature of homogenization is important, particularly as it relates to the viscosity of the dispersed phase. Banks et al. (1981b) used a homogenization temperature of 55 °C in the two pass pilot scale production of cream liqueurs at a total homogenization pressure of 27.6 MPa (4000 psi), and subsequently Muir and Banks (1986) used 50 °C in a multiple homogenization of cream liqueurs investigation.

Many applications require a very uniform droplet size distribution in the emulsion, either for control of creaming rate or for some physical action or characteristic required of the emulsion. This can be accomplished in the homogenizer by passing the product more than once through the valve (Pandolfe and Kinney 1983). Multiple passes do not reduce the modal particle size but reduce the probability of having oversized particles thereby leading to a narrower distribution of sizes. A single pass process minimizes production time and maximizes product consistency but for an extremely uniform particle size as discussed above or for products such as cream liqueurs which require a very small (0.1–0.3 μm) average particle size, it is simply not possible to reach these goals in a single pass through a homogenizer (Masucci 1989b).

If the average particle size is greater than the critical particle size, then the homogenizing pressure should be increased. On the other hand, if there is sizeable tail of particles greater than the critical diameter or a bimodal distribution where the second peak occurs at a particle size in excess of the critical diameter, then the number of passes should be increased (Masucci 1989c). This rule of thumb assumes that the premix quality is good, the formulation is correct and there are no mechanical deficiencies (Masucci 1989c).

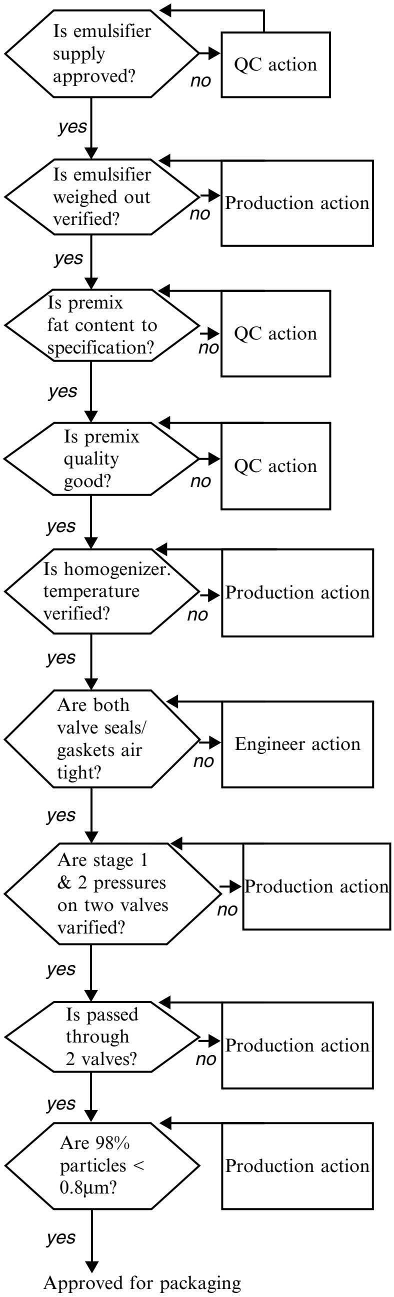
In production of stable, high-quality traditional Irish Cream liqueurs by one manufacturer (Fig. 4.1), the optimal homogenization temperature and total pressure and number of passes are considered to be 57 °C, 27.6 MPa (4000 psi) and two, respectively. Whilst the case for two passes is well established, there may well be a case to gain energy savings through the use of a lower homogenization temperature and total pressure of 55 °C and 24.1 MPa (3500 psi), respectively, without detriment to the traditional Irish Cream liqueur stability and quality.

4.3.5 Troubleshooting Protocol

A troubleshooting protocol for the production of traditional Irish Cream liqueurs can be specified (Fig. 4.2) based on the scientific and technical literature review and observations at one manufacturer.

4.4 Conclusions

Traditional Irish Cream liqueurs are added value, long-life, oil-in-water emulsions, combining the flavour of an alcoholic drink with the texture of thickened cream. The fundamental studies of a number of workers in the UK and Ireland during the 1980s have enabled the significant commercial problems associated with the production of Irish Cream liqueurs to be overcome. Effective classical high-pressure homogenization and selection of sodium caseinate, either as the sole stabilizer at a protein to fat ratio of *circa* 0.2 or combined with glycerol monostearate at a reduced level, can prevent creaming and fat plug formation. Addition of tri-sodium citrate di-hydrate at 0.19 % by weight in the formulation can prevent emulsion destabilization by calcium-induced aggregation and minimize calcium citrate crystal deposits in an exemplar Irish Cream liqueur. The standard two-stage design “plug-type” homogenizer valve is widely used in the production of Irish Cream liqueurs. For efficient homogenization operation, it is important to achieve a good premix quality, exclude air, control the dispersed phase viscosity and effectively specify homogenization parameters. The use of a high-shear in-line mixer within the process improves premix quality. A homogenization temperature of 55 °C helps to control the dispersed phase viscosity. Effective homogenization results through the use of a total pressure in two stages, typically at 24.1 MPa (3500 psi) and 3.45 MPa (500 psi), and over two homogenization passes. There may be a case for a lower total homogenization pressure (24.1 MPa; 3500 psi) based on energy savings, and the use of a troubleshooting protocol for the production of traditional Irish Cream liqueurs. The commercial importance of cream-based liqueurs will continue to drive scientific and technical developments aimed at lowering the cost of manufacture, reformulating existing products and generating innovative new products.



Action	*Comment*
Use sample per batch to prepare a standard lab liqueur. If 98% are not <0.8µm then reject.	Wrong type or inactive emulsifier leads to coalescence or breaking. (↑ Pressure to detect)
Signed off by operator.	Insufficient emulsifier leads to coalescence.
Adjust fat to within specification.	Slight increase in mean particle size at higher fat.
Particle size for premix. If 70% are not < 2µm then recirculate.	Poor premix will result in larger mean particle size.
Signed off by operator	Low temperature increases fat viscosity resulting in larger mean particle size.
Signed off by engineer.	Inclusion of air results in creaming shortly after homogenization. (↑ Pressure to detect)
Signed off by operator.	2nd stage increases efficiency and minimizes clumping.
Signed off by operator.	Needed to reduce probability of oversized particles.
Pass through again. Use higher pressure if mean particle size is >0.4µm.	Higher pressure should make overworking detectable. Rework if not in specification

Fig. 4.2 Troubleshooting protocol for the production of traditional Irish Cream liqueurs

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