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Turbidity in oil-in-water-emulsions — Key factors and visual perception

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ABSTRACT

The aim of the present study is to systematically describe the factors affecting turbidity in beverage emulsions and to get a better understanding of visual perception of turbidity. The sensory evaluation of the human visual perception of turbidity showed that humans are most sensitive to turbidity differences between two samples in the range between 1000 and 1500 NTU (ratio) (nephelometric turbidity units). At very high turbidity values >2000 TU in NTU (ratio) were needed to distinguish between samples that they were perceived significantly different. Particle size was the most important factor affecting turbidity. It was shown that a maximum turbidity occurs at a mean volume - surface diameter of 0.2 μm for the oil droplet size. Additional parameters were the refractive index, the composition of the aqueous phase and the presence of excess emulsifier. In a concentration typical for a beverage emulsion a change in the refractive index of the oil phase may allow the alteration of turbidity by up to 30%. With the knowledge on visual perception of turbidity and the determining factors, turbidity can be tailored in product development according to the customer requirements and in quality control to define acceptable variations in optical appearance.

1. Introduction

The degree of turbidity is an important parameter reflecting the quality of a cloudy fruit juice (Kolniak-Ostek, Oszmiański, & Wojdyło, 2013). Therefore the visual appearance of a cloudy drink is a critical factor for consumer acceptance (Mirhosseini & Tan, 2010). Turbidity in fruit-derived beverages gives a natural appearance, and is achieved through addition of so-called clouds or clouding agents to the majority of non-alcoholic beverages (Espachs-Barroso, Soliva-Fortuny, & Martín-Belloso, 2005; Mirhosseini, Tan & Taherian, 2008; Shachman, 2004; Taherian, Fustier, & Ramaswamy, 2006).

A cloud provides turbidity, flavor, aroma, mouthfeel and optionally color and is typically added in concentrations of 0.01–0.2 wt% of the final product (Buffo, Reineccius, & Oehlert, 2001; Dickinson, 1994; Espachs-Barroso et al., 2005; Harnsilawat, Pongsawatmanit, & McClements, 2006; Tan, 1998). At this dosage turbidity usually ranges between 100 and 250 NTU (ratio). Clouds are concentrated oil-in-water emulsions in which the turbidity results from the scattering of light by dispersed oil droplets with oil droplet size between 0.5 and 5 μm (Dickinson, 1994; Tan, 1998). In this context the scattering of light is governed by the size and concentration of the dispersed phase, the ratio of refractive index (RI) between disperse and continuous phase and the wavelength and angle of the incident light (Kleizen et al., 1995). Physical background of the scattering of light has been reviewed extensively e.g. in Mie, 1908; Bailey, Nichols, & Kraemer, 1935; Oster, 1948; Lothian & Chappel, 1951; Walstra, 1965; Kerker, 1969; Van de Hulst, 1981; and Bohren & Huffman, 1983.

Concerning its composition a cloud is most commonly stabilized by amphiphilic polysaccharides, such as gum Arabic or hydrophobically modified starch (Buffo, Reineccius, & Oehlert, 2002; Chanamai & McClements, 2001). The oil phase usually consists of vegetable oil, flavor oil, and a weighting agent (Tan, 1998). Weighting agents increase the density of the oil phase and therefore contribute to the stabilization of an emulsion through minimization of the difference in density between the different phases. A typical weighting agent is the glycerol ester of wood rosin (glyceryl abietate) (Given, 2009; Lim et al., 2011). With respect to the final application it needs to be mentioned that the aqueous phase of a beverage emulsions is normally acidic with a pH below 3 (Harnsilawat et al., 2006). A finished beverage typically contains additional sugar (10°Brix) and/or sweetener, citric acid and preservative (Reiner, Reineccius, & Peppard, 2010; Tan, 1998).

In the past turbidity measurement was mainly used as a tool to characterise clouding agents and cloud stability. The latter is the most critical quality parameter of a beverage emulsion is its stability in the diluted state (Kolniak-Ostek et al., 2013; Cao et al., 2012: Rao & McClements, 2012; Ibrahim et al., 2011; Reiner et al., 2010; Taherian et al., 2006; Harnsilawat et al., 2006; Liang et al., 2006; Dłużewska, Panasiewicz, & Leszczyński, 2004; Beveridge, 2002; Cameron, Baker, & Grohmann, 1997; Sreenath, Crandall, & Baker, 1995; Morris, 1987; Kaufman & Garti, 1984; Ray, Johnson, & Sullivan, 1983; Herrera, Matthews, & Crandall, 1979). In contrast, only little work has been done on the

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relationship between the human visual perception of turbidity and instrumental turbidity measurement. Few studies are available, in which minimum thresholds of the perception of turbidity were determined (Carrasco & Siebert, 1999; Fleet & Siebert, 2005, 2006; Horne, Olabi, Greenwalt, & Lawless, 2001 and Malcolmson, Jeffery, Sharma, & Ng, 1989). Malcolmson et al. (1989) examined the relationship of human perception of apple juice and instrumental turbidity values. He reported that a change in clarity was only observed by the sensory panel at a large difference in instrumental reading. This may be due to the fact, that turbidity was measured at a 90° detection angle which is very sensitive for small particles but which contribute only little to perceived turbidity. Another important factor might be the color of apple juice. Carrasco and Siebert (1999) found turbidity thresholds visible for the human eye below 1 NTU, but under very controlled viewing conditions with a defined light source and a box with a black background. Horne et al. (2001) on the other hand measured visual haze detection thresholds for differently colored emulsions under less controlled conditions to simulate a consumer situation. The thresholds with up to 4.5 NTU[1] reported are much higher than the results previously reported by the group of Carrasco and Siebert (1999), but overall are still very low. Furthermore, Carrasco and Siebert (1999) investigated the influence of color on the human visual perception of turbidity. The authors found that higher particle concentrations were needed to detect haze in red-colored suspensions compared with yellow-colored or clear suspensions. Fleet and Siebert (2005) compared in a similar study clear, caramel- and dark brown-colored suspensions and found thresholds between 0.2 and 2.2 NTU with the lowest values for clear and the highest results for dark brown-colored samples. As turbidity measurements were operated with white light, the readings were influenced by the color as well and might have produced more pronounced differences at NIR readings. The latter can be concluded from a study by Hongve and Åkesson (1998), who compared white light measurements against readings in the NIR region at 860 nm, and found that absorption due to color present reduces NTU (ratio) readings. On the other hand, at high turbidity levels saturation may occur and panelists are possibly not able any more to distinguish different levels of turbidity. Carrasco and Siebert (1999) concluded from a magnitude estimation study that the panelist response became saturated at higher turbidities above 500 NTU. Anyhow no literature on the question, what difference in turbidity may be distinguished by human visual perception at a specific level of turbidity, is available. This holds particularly true for very high levels of turbidity up to 10.000 NTU as they may occur in cloudy beverages and juice concentrates.

From the literature outlined above it becomes evident that the correlation of human visual perception and instrumental reading is of major interest in consumer studies and the beverage industry. On the other hand studies have been performed to optimize beverage clouding systems (Gharibzahedi, Mousavi, Hamedi, & Ghasemlou, 2012; Klein, Aserin, Svitov, & Garti, 2010; Mirhosseini, Tan, Aghlara, et al., 2008; Mirhosseini, Tan, Hamid and Yusof, 2008; Mirhosseini, Tan, Hamid, Yusof, & Chern, 2009; Mirhosseini, Tan & Taherian, 2008). Since these studies did not focus on turbidity, the factors affecting this parameter are still poorly understood. Therefore the aim of the present study was to systematically describe the factors affecting turbidity in beverage emulsions and to get a better understanding of consumer perception of turbidity. From an industrial perspective, with this data the development of a beverage can be more efficiently directed towards the desired optical appearance. To fulfill this aim the impact of particle size, particle concentration, type of oil phase and type of emulsifier on turbidity were investigated for a polydisperse emulsion system. In addition, the instrumental turbidity measurement was correlated to the human visual perception via sensory analysis.

[1] Nephelometric turbidity units - NTU

2. Materials and methods

The following materials were purchased or kindly provided by the suppliers mentioned below: Gum Arabic[2] Senegal type (GA, Nexira GmbH, München, Germany), medium chain triglyceride[3] (MCT, CREMER OLEO GmbH & Co. KG, Hamburg, Germany), orange oil[4] (OO, Citrosuco, Matão, Brazil), glycerol ester of wood rosin,[5] beverage grade (EG, Pinova, Brunswick NJ, USA), α-tocopherol (BASF, Ludwigsburg, Germany), whey protein concentrate[6] 80% (WPC, FrieslandCampina DMV bv, Veghel, Netherlands), octenyl-succinate derivatised starch[7] refined from waxy maize, *Quillaja saponaria Molina* extract[8] (both National Starch & Chemical Ltd., Manchester, UK), potassium sorbate (Eastman Chemical Company, Kingsport, USA), citric acid (Jungbunzlauer Suisse AG, Basel, Switzerland), 72.7°Brix inverted sugar syrup (Nordzucker AG, Braunschweig, Germany).

To fulfill the aim of the study, different sets of experiments were performed.

2.1. Emulsion preparation

A standard emulsion was prepared by dissolving gum Arabic (13.5%w/w) and preservatives in water with RW20DZM agitator with R 1302 dissolver stirrer (IKA®-Werke GmbH & Co. KG, Staufen, Germany) and left over night for hydration. MCT (7.15% w/w) was gradually added to the continuous phase while mixing with Ultra-Turrax T50 with dispersion unit S50 N (IKA®-Werke GmbH & Co. KG, Staufen, Germany) at 6000 rpm for 5 min to generate a pre-emulsion[9] (PE). The PE was left at least 2 h for deaeration before homogenization with a laboratory table top model homogenizer NS1001L2K (GEA Niro Soavi S.p.A., Parma, Italy). Unless stated otherwise, homogenization was done in three cycles in a two stage process at 20/5 MPa. Concentrations for analytical purposes were made with deionized water. To estimate the error in multiple preparation in emulsions the standard emulsion was prepared in triplicate resulting in a particle size of $\overline{x}_{1,2}$ = 0.3 μm ± 0.01 standard deviation and turbidity being 119.8 NTU(ratio) ± 6.92 standard deviation.

2.2. Impact of particle size

To analyze the impact of particle size on turbidity emulsions with constant oil content but different particle size distributions had to be prepared. This was achieved by varying the homogenization pressure during emulsification of the standard emulsion. A first sample was taken after pre-emulsification. The PE was then divided into four equal portions, which were homogenized in three cycles at varying pressures (8/3 MPa, 20/5 MPa, 35/5 MPa, 50/5 MPa). At each pressure stage a sample was taken. A concentration of the respective emulsion (0.2%) was prepared for each sample prior to turbidity analysis and particle size measurement.

2.3. Impact of particle amount

At constant oil content, a decrease in particle size leads to a significant higher amount of oil droplets. To evaluate whether the amount of particles has an impact on turbidity, particle count calculations were undertaken for two emulsions with a distinct difference in the particle size distribution. Calculations were performed using the standard emulsion

[2] Gum arabic - GA
[3] Medium chain triglyceride - MCT
[4] Orange oil - OO
[5] Glycerol ester of wood rosin - EG
[6] Whey protein concentrate - WPC
[7] Octenyl-succinate derivatised starch - OSA
[8] *Quillaja saponaria Molina* extract - Q
[9] Pre-emulsion - PE