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The morphology of salt crystals affects the perception of saltiness

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ABSTRACT

High intake of salt (NaCl) has been associated with risk of non-communicable diseases, including hypertension, cardiovascular disease and stroke. Several strategies for reducing salt in foods are under study, including the relation of crystal morphology on dissolution properties of salt in the mouth. The aim of this paper was to study the dissolution of salt crystals with different morphologies in artificial saliva and to correlate the findings with the perception of saltiness over time. The morphology of five commercial salts was analyzed by scanning electronic microscopy and micro-CT studies. Shape parameters of crystals were determined using images from an optical microscope. Crystal dissolution in artificial saliva was evaluated using video-microscopy and the perception of saltiness was evaluated using sensorial test of time–intensity at standardized sodium content. Salt morphology was correlated well with dissolution rate and certain time–intensity parameters (time to maximum intensity, intensity at maximum and increase angle). Non-cubic and agglomerated crystals, such as Kosher and Maldon salts, were dissolved faster (dissolution rate up to 3.8 times higher) and experienced maximum saltiness (up to 17% more) at shorter times (up to 40% less). Crystal morphology may be a variable to consider to achieve sodium reduction while maintaining salt intensity.

1. Introduction

High sodium intake has been associated with a high risk of non-communicable diseases (NCDs), including hypertension, cardiovascular disease and stroke. NCDs are the main contributor to mortality and morbidity in the world. Reducing salt intake, the main source of sodium in the diet, could therefore be an important target for improving public health (Bibbins-Domingo et al., 2010; WHO, 2003, 2012). The maximum level of sodium intake recommended by the World Health Organization is 2 g per day for adults (≥16 years old) equivalent to 5 g of salt (NaCl) per day (WHO, 2003, 2012). However, most people consume too much salt, on average between 9 and 12 g per day, or around twice the recommended maximum level of intake (WHO, 2014).

The main source of sodium in the diet comes from salt that is added to foods. In the USA and Europe, between 75 and 80% of salt intake comes from processed foods (Brown, Tzoulaki, Candeias, & Elliott, 2009; Mattes & Donnelly, 1991). Therefore, it is necessary for the food industry to implement strategies to reduce the content of salt.

Several strategies have been developed by the industry to reduce the level of sodium in foods while maintaining the overall quality and acceptability. Most of these are strategies focused on cognitive mechanisms (increasing consumer awareness of preferring low sodium foods) and chemical approaches (using ingredients to increase the perception of true saltiness). The option of designing structures to optimize the perception of saltiness has received lesser attention (Busch, Yong, & Goh, 2013). It emphasizes the control of the release of salt from food matrices, its delivery in the oral cavity, and the generation of saltiness perception (Kuo & Lee, 2014). Thus, this strategy involves the improvement of the dissolution rate of crystals from dry foods, the spatial distribution of salt in food matrices, and the textural effects on the food matrix (Busch et al., 2013).

Phan et al. (2008) showed that between 70 and 95% of sodium (or salt) may remain in the food matrix after swallowing. With regard to salt crystals applied topically to dry foods (i.e. potato chips), Tian and Fisk (2012) suggest that a significant proportion of sodium can be swallowed without being perceived, because saltiness is not perceived when the sodium is delivered after swallowing. Ideally most sodium in the matrix should become solubilized and contribute to the perception of saltiness.

There is a relationship between the dissolution rate of salt crystals and perception of saltiness. Salt crystals that have a greater surface area are dissolved faster. Increasing the surface area of a crystal can be achieved with a smaller crystal size, a hollow shape, or a crystal structure that can be fractionated during dissolution (Jensen, Smith, Fear, Schimoeller, & Johnson, 2011; Quilaqueo & Aguilera, 2015; Rama et al., 2012). However, the influence of characteristics of different salt types, specifically different morphologies, origin, composition, on

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dissolution rate and perception of saltiness, remains relatively unstudied (Quilaqueo & Aguilera, 2015; Vella, Marcone, & Duizer, 2012). The aim of this paper is to study the dissolution of salt crystals with different morphologies using artificial saliva, and to correlate the results with the perception of salt intensity over time.

2. Materials and methods

2.1. Materials

Five varieties of commercial salts were used in this work (Table 1), selected by their morphology. A uniform size fraction of crystals was isolated between sieves number 20 and 14 (0.850 and 1.41 mm of aperture respectively) by manually sieving 200 g of each salt variety for 2 min.

2.2. Salt characterization

2.2.1. Mineral analysis

Minerals, sodium, calcium, magnesium, phosphorous, potassium, sulfur, and iron in salt crystals were determined by inductively coupled plasma optical emission spectrometry (VISTA-pro, Varian Canada, Mississauga, ON, Canada). Samples were evaluated in duplicate.

2.2.2. Moisture content

The moisture content of the salt crystals was obtained through an infrared moisture analyzer (MA 30, Sartorius, Göttingen, Germany) working at 130 °C until constant weight of sample. The results are expressed on wet basis of triplicates.

2.2.3. Apparent density

The apparent density (ρ_{app}) was determined by the equation $\rho_{app} = m_s/V_{app}$ where m_s is the mass of dry solids (kg) and V_{app} corresponds to the apparent volume (m^3). The mass of the samples was determined in a UX620H analytical scale (Shimadzu, Philippines). The apparent volume was obtained by liquid displacement in a glass pycnometer (accuracy of 0.05 mL) after immersing the samples in n-heptane at ambient temperature (about 25 °C). Results were based on four replicates (Oikonomopoulou, Krokida, & Karathanos, 2013).

2.2.4. Scanning electron microscopy and micro-computed tomography

Samples of salt were assessed by scanning electron microscopy (SEM) (Hitachi S 570, Hitachi High Technologies, Tokyo, Japan) at an accelerating potential of 10 kV. Salt crystals were placed on a specimen stub and coated with gold/palladium. At least triplicate specimens were viewed at several magnifications.

In order to examine qualitatively the external and internal structure of crystals qualitatively, micro-computed tomography (micro-CT) was done. Micro-CT scanning was carried out in single crystal (at least one of each sample) using Skyscan 1272 (Bruker MicroCT, Kontich, Belgium). The 3D reconstruction was done with the software programs called NRecon and CTvox (Bruker microCT, Kontich, Belgium).

2.2.5. Optical microscopy

The morphology of salt crystals was assessed by image analysis of images obtained by optical microscopy. Crystals deposited on a glass slide were observed under a stereo microscope (SMZ 2B-2T, Nikon Corp., Japan) and images were acquired with a digital camera ToupCam (Ucmos08000 KPA, Touptek Photonics, China) coupled to the microscope and using ToupView 3.5 software (ToupTek, Zhejiang, China). The images obtained were processed and analyzed using ImageJ 1.45s software (National Institutes of Health, USA).

2D shape and size descriptors were determined, based on image analysis. The descriptors were: projected area (A); equivalent diameter ($D_{eq} = 2\sqrt{A/\pi}$); circularity ($C = 4\pi(A/P^2)$) where P corresponds to perimeter; Feret diameter (F) distribution, from which the maximal (F_{max}) and minimal (F_{min}) were determined; elongation ($E = F_{max}/F_{min}$); and aspect ratio (AR = Major axis/Minor axis of fitted ellipse) (Ferreira, Faria, Rocha, Feyo de Azevedo, & Lopes, 2005; Ferreira & Rasband, 2012).

2.3. Crystal dissolution in artificial saliva by video-microscopy

The dissolution of single crystals in artificial saliva at 37 °C was assessed by image analysis of video-microscopy images as described in Quilaqueo and Aguilera (2015). The artificial saliva was composed by 5.208 g of $NaHCO_3$, 1.045 g of K_2HPO_4, 0.877 g of NaCl, 0.477 g of KCl, 0.441 g $CaCl_2.2H_2O$ and 2.160 g of mucin (porcine pancreas mucin, Sigma-Aldrich, St. Louis, MO, USA) dissolved in 1 L of deionized water. NaN_3 was added at a concentration of 0.5 g/L to prevent microbial growth (Van Ruth, Grossmann, Geary, & Delahunty, 2001).

The acquisition of images during crystal dissolution was performed as described before for single crystals. Recording began when a single particle of salt was placed into a glass capsule. Immediately after artificial saliva (500 μL) at 37 °C was added and temperature was maintained through a hot-stage system (Quilaqueo & Aguilera, 2015). Recorded images were processed and analyzed with ImageJ 1.45s software (National Institutes of Health, USA) and each salt type was tested at least in quintuplicate.

The dissolution rate was calculated based on the reduction of the projected area of crystals over time and the dissolution kinetics were fitted to a model. Zero order (linear) and first order (non-linear) models were used (Quilaqueo & Aguilera, 2015), according to the following equations: $A_t = -K_0 \cdot t + A_0$ for zero order, where A_t is the crystal area (as percentage of the initial area) at time t (s), K_0 (s^{-1}) is a constant related to the dissolution velocity and A_0 is the initial crystal area and; $ln(A_t/A_0) = K_1$ for the first order model, where K_1 (s^{-1}) is a constant related to the dissolution velocity (Costa & Sousa, 2001).

2.4. Sensory evaluation

Time–intensity (TI) analysis was performed to evaluate salt intensity of commercial salts perceived in mouth over time. The procedures of the sensory TI test were first approved by the University of Guelph Ethics Review Board (REB number 14AU014). Eleven panelists, not allergic or sensitive to salt, food preservatives, iodine and anticaking agents present in commercial salts, with experience in sensory evaluation were selected for the sensory panel. The panelists were trained

Table 1
Description of commercial salts analyzed and amount of salt containing 20 mg of sodium (used for sensory testing).

Salt type	Description	Supplier	Salt amount (g)
Brittany Grey	Grey sea salt free of artificial additives	Drogheria e alimentari SpA, France	0.056
Extra Coarse	Iodized salt from rock deposits	SLP salt, Chile	0.053
Kosher	Diamond crystals free of artificial additives	Cargill Inc., USA	0.052
Maison Orphée	Grey sea salt free of artificial additives	La Maison Orphée Inc., France	0.055
Maldon	Sea salt free of artificial additives	Maldon Salt, UK	0.049