Is all Fleur de sel the same? Experience from artisanal saltworks in Castro Marim, Portugal

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Salt Production

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Abstract

We describe production of Fleur de sel in the solar saltponds of Castro Marim (Portugal) where the local producers reoriented and substantially increased output of this variety of salt. Indeed this culinary specialty is commercialized for a price order of magnitude higher than coarse marine salt which traditionally dominated the production. Because of a complete lack of published information on that subject the present study aims to elucidate factors, which influence the quality and characteristics of the end product. Crystals size, and density of Fleur de sel samples, collected during the period of 70 days in an artisanal salt unit, were studied. The study resulted in a classification of 3 types of crystals according to their size. Category A was attributed to the biggest crystals, with surface ranging from 18 to 24 mm^2, considered as the best quality crystals taking into account market requirements. Their formation took place in the following atmospheric conditions: air temperature of 29.6 ºC; relative humidity of air 40.3%; southern wind direction and speed of 6.0 knots; solar radiation of 31,227 KJ/m^2; and, an evaporation rate of 1.38 mm/day. Main physicochemical parameters of the brine measured during the formation hours of the biggest crystals were significantly different from those prevailing during the formation of other two categories of crystals. Typical values of the measured parameters corresponding to the best quality crystals were: surface electric conductivity (EC) of 215.32 mS/cm and bottom EC of 216.76 mS/cm; surface temperature of 37.16 ºC and bottom temperature of 37.28 ºC. The worst quality (C) formed at lowest values of EC and temperature with a mean surface and bottom EC of 200.15mS/cm and 196.86mS/cm, respectively; and 32.45 ºC and 32.75 ºC surface and bottom temperatures, respectively. Moreover, during the formation of category A crystals the difference between surface and bottom temperature was the lowest and bottom EC was higher than surface EC. 1 gram of category C crystals contained 156.51 (13.59 St Dev) particles, while category A samples contained 96.73 (7.11 St Dev) particles. The former results should be considered when focusing on the production of Fleur de sel, by adjusting some of the described parameters, such as the brine characteristics according to the weather forecast or on a real time basis.

Introduction

Castro Marim is a small Portuguese town situated in the Guadiana River Estuary which makes the border with Spain. The Guadiana River is situated in the southwest of the Iberian Peninsula with a total length of 820 km and a catchment area of 67,000 km^2. The lower estuary resulted from Holocene sediment infilling of the river paleovalley and progradation of the delta; extensive salt marshes developed on the accreted sediments surface at both sides of the main channel (Boski et al., 2008). The region has a Mediterranean climate, experiencing mild winters and hot summers. During summers, when freshwater discharge from the river is at a minimum, the estuary is tidally mixed and has a strong horizontal salinity gradient (Guimarães et al., 2012). During the rainy winters when the discharge is at its peak, salt wedge and pronounced vertical salinity gradient are observed. The annual average air temperature varies from 14ºC to 18°C, 80% of the precipitation occurs during autumn and winter with a mean yearly rainfall of 400 to 600 mm (Chícharo et al., 2001) and the potential evaporation is typically 2-3 times higher than the latter figure. Castro Marim landscape is basically shaped by saltworks and tree plantations. Salt production in the Iberian Pyrite Belt have been practiced since Phoenician times (Delgado et al., 2011). The present study is focused on Fleur de sel (or Flower of salt) crystals produced in one of these artisanal saltworks. The study looks into the influences of weather conditions and brine physico-chemistry in the formation of Fleur de sel crystals, using the same production method.

Fleur de sel form initially as two-dimensional crystals of sodium chloride evolving to hopper pyramids. These tetragonal hollow pyramids float and grow as thin layers on the high-density brine surface of the crystallizers (shallow ponds containing brine at saturation). Due to the fact that only one face of the cube is attached to the surface, growth of floating thin tabular crystals takes place initially along their edges. Progressively the third dimension develops downwards from surface, changing the tabular flakes into inverted hollow Mayan pyramids (Fontana et al., 2011) which under their own weight, through gravitational sedimentation, are deposited at the crystallizer's bottom. The collection/harvesting of Fleur de sel should intervene before the deposition of crystals. Overall, it is known that variations in salt origin, brine source and manufacturing specifications result in sea salts with unique chemical composition, grain size, and crystal shape and colour (Vella et al., 2012). Among the scarce literature on this topic, the experiences of Varnavas and Lekkas (1996) concluded that the meteorological conditions of a monitored area, combined with other environmental conditions, played an important role in the final quality of the salt produced. They examined the meteorological data affecting a saltpond and showed that winds blow from the North during most of the year; this N-S direction zone coincided with the zone of the finest sediments and the less clean coarse salt produced.

There were several attempts to characterize the conditions in which pyramidal shaped hopper crystals are formed. Dellwig (1955), observed formation temperatures in the interval 32.0 to 48.8ºC. Fontana et al. (2015) efforts to grow Fleur de sel crystals on the International Space Station were unsuccessful as it was concluded that the required conditions of solution temperatures above 25 ºC at 40% relative humidity were not attained.

Although not considered in this study, microorganisms play some role in solar salt production. Microorganisms prevent brine leakage by forming mats in the bottom of the ponds, and they also produce coloured cells that increase solar energy absorption which improves evaporation (Davis, 1973). *Dunaliella salina* cells have been found "to be directly connected to alterations of

NaCl crystalline structure and with the quantity and quality of contaminants in the salt" (Giordano et al., 2014).

Methods

Two crystallizers, part of an 1 ha artisanal saltpond, were monitored for 70 days during salt harvesting season. Brine conductivity and temperature were measured every hour approximately from 9 a.m. to 6 p.m. using the multiparameter Hanna Instruments HI98192 (accuracy of EC: ±1% of reading; and temperature: ±0.2ºC). Fleur de sel was collected manually from the brine surface at around 6.00 p.m. and sampled after decanting and sundrying for 2 days. At the same time, an in-situ weather station (Oregon® professional WMW200) recorded, every 10 minutes, the following parameters: air temperature and relative humidity, precipitation, wind direction and speed, and pressure. Solar radiation data was provided by the Portuguese Sea and Atmosphere Institute (Instituto Português do Mar e da Atmosfera). Evaporation rate was calculated following Penman equation.

Salt crystals were counted and measured and photographed (Fig.1) in order to classify them.

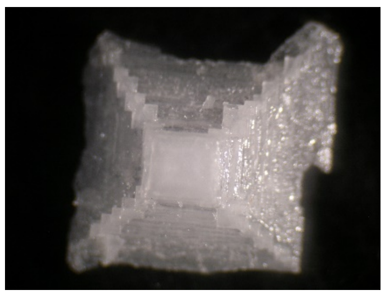

Figure 1: Morphological detail of a Fleur de sel crystal

For each Fleur de sel sample, 3 replicates of 1 gr were weighted using Kern EG620-3NM (e=0.01gr) scale, and they were photographed with the Olympus Camedia C-7070 (7.1 megapixels; 4x zoom) camera. Measurement and counting of Fleur de sel crystals was carried out through image analysis, using ImageJ software. For an automatic particle counting and crystal area measurement with the former software, the filter was adjusted by comparisons of manual particle counts to software particle counts of a randomly selected region of the images (Fig. 2).

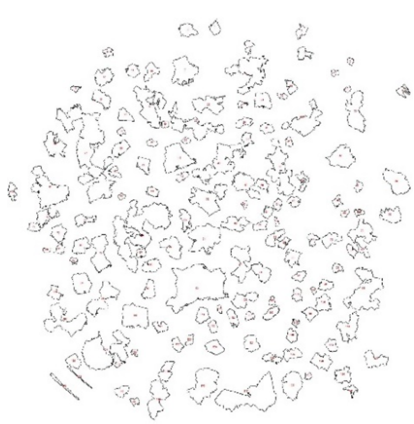

Figure 2: Particles counting with software ImageJ for a 1 gram sample of Fleur de sel

Meteorological data and brine characteristics recorded during formation days were analysed to define the main parameters influencing each crystal type formation.

Results
Sea salt samples representing 38 harvesting days were analysed. Average number of crystals and their mean size are reported in Table 1.

	Crystallizer 1		Crystallizer 2	
Date	**Average number of crystals/gr ± St Dev**	**Average size (mm^2) ± St Dev**	**Average number of crystals/gr ± St Dev**	**Average size (mm^2) ± St Dev**
13-Jun	91±7.5	13.7±1.6		
17-Jun	102±11.7	19.3±2.5		
18-Jun	89±1.5	19.6±0.7	105±4.2	15.3±0.6
19-Jun	94±5.2	19.8±5.2	109±2.0	18.8±0.4
22-Jun	69±8.7	23.8±2.2		
25-Jun	111±13.1	20.9±2.2	119±24.0	16.9±3.5
26-Jun	112±7.0	16.2±1.1		
27-Jun	107±11.0	18.6±1.0	111±8.7	19.8±1.6
29-Jun	94±2.1	20.1±1.2	80±7.0	21.8±2.3
01-Jul	153±10.1	10.8±1.5	160±28.4	9.7±1.3
02-Jul	184±20.4	9.5±1.2	160±12.1	12.2±0.9
04-Jul	121±21.2	14.9±3.5	166±16.5	10.6±1.9
08-Jul	142±8.0	10.6±0.3		
09-Jul	122±10.2	13.9±1.1	161±26.8	11.6±1.8
10-Jul			146±6.6	11.6±0.4
11-Jul			162±18.4	10.6±0.8
12-Jul	171±22.4	10.2±1.2	157±2.1	9.6±0.8
13-Jul	155±7.6	11.3±1.1		
14-Jul	157±3.1	11.3±0.9		
16-Jul			108±19.3	15.9±3.0
18-Jul	150±19.1	12.4±1.2	160±15.7	9.8±1.0

19-Jul	109±12.7	15.5±1.0		
20-Jul	109±24.7	15.6±1.2	192±2.5	10.4±1.0
21-Jul			149±5.9	11.7±1.2
22-Jul	105±13.2	15.0±4.6		
23-Jul	126±11.2	10.3±3.2		
24-Jul			125±28.3	16.1±3.8
25-Jul			126±9.1	8.2±1.5
26-Jul	115±26.9	12.6±4.9	95±6.7	11.4±1.0
27-Jul	154±12.7	8.6±0.6		
29-Jul	161±9.5	10.0±0.7	148±5.2	10.9±1.0
30-Jul	137±14.6	11.9±1.4	160±14.5	9.8±0.7
04-Aug	151±29.6	11.2±1.8		
05-Aug	176±22.9	10.1±1.4		
06-Aug	189±6.2	8.4±0.4	179±14.7	11.9±1.3
11-Aug			167±9.8	9.0±0.6
12-Aug			212±23.3	7.0±0.8
13-Aug	116±23.2	12.2±1.8		

Table 1: Number of crystals per gram and crystal size for each collection day per crystallizer (blank=no production)

Crystals were divided into 3 categories according to the following mean size thresholds (mm^2): from 7 to 13, type C; from 13 to 18, type B; and from 18 to 24, type A. These thresholds are adopted based on market requirements, where three types of Fleur de sel could be differentiated depending on the size and use. Type C crystals have the lowest commercial interest due to its small size and it is generally sold mixed with other gastronomic ingredients (i.e. herbs, lemon); whilst type A crystals have the highest economic value, usually commercialised under the name of “salt flakes”. Commercialised salt flakes are usually produced using anthropogenic energy and different facilities. Table 2 shows the number of particles per gram according to this classification.

	Crystal C	Crystal B	Crystal A
Mean number of particles ± St Dev	156.51 ±13.59	113.81 ±17.85	96.73 ±7.11

Table 2: Number of crystals per gram according to crystal classification (both crystallizers)

The monitored meteorological parameters were related to the crystal size using descriptive statistics (Table 3). Precipitation is not included, as it was recorded only during 2 unproductive days.

	Crystals C	Crystals B	Crystals A
Mean temperature (ºC)	29.84	30.04	29.57
Mean Relative Humidity (%)	40.39	39.26	40.28
Mean wind direction (º)	222.2	210.3	184.8
Mean wind speed (Knots)	8.68	8.27	6.01
Solar radiation (KJ/m^2)	29,297.80	29,356.12	31,226.94
Evaporation (mm/day)	1.74	1.58	1.38

Table 3: Meteorological parameters related to crystal category (both crystallizers). Meteorological parameter values refer to the mean of the measurements taken every 10 minutes from 9 a.m. to 6.00 p.m.

Crystals categories were described according to brine EC and temperature. EC and temperature measurements were carried out in two different sample points of the crystallizers every hour from 9 a.m. to 6 p.m., approximately. Table 4 shows mean values of surface and bottom EC and temperature obtained from 8 sample points (4 at each crystallizer: 2 at the surface and 2 at the bottom).

	Crystals C	Crystals B	Crystals A
Surface EC (mS/cm)	200.15	203.89	215.32
Surface temperature (ºC)	32.45	33.65	37.16
Bottom EC (mS/cm)	196.86	202.73	216.76
Bottom temperature (ºC)	32.75	33.90	37.28
EC difference (mS/cm)	3.29	1.16	-1.45
Temperature difference (ºC)	-0.30	-0.25	-0.12

Table 4: Brine characteristics related to crystal type (both crystallizers)

Discussion

Results point to the existing relation between meteorological conditions and crystals size. The A crystals, ranging in surface from 18 to 24 mm^2, were formed under air temperature of 29.6 ºC; air relative humidity of 40.3 %; southern wind direction and speed of 6.0 knots; solar radiation of 31,227 KJ/m^2; and, an evaporation rate of 1.38 mm/day. The former results, compared to the other two categories of crystals, showed that the commercially most interesting crystals are formed under the highest solar radiation, and under the lowest wind speed and evaporation rate. Category A formation occurred on days with prevailing southern (185º) winds direction, compared to the southwestern winds (222.2º) and 8.68 knots of average speed that shaped category C crystals.

Brine concentrations and temperatures, corresponding to the typical conditions of A crystals formation, showed the highest values when compared to the conditions typical for other two categories: surface and bottom EC of 215.32 mS/cm and 216.76 mS/cm, respectively; surface and bottom temperature of 37.16 ºC and 37.28 ºC, respectively. The smallest crystals (C) were formed under the lowest surface and bottom EC of 200.15mS/cm and 196.86mS/cm, respectively; and lowest surface and bottom temperatures of 32.45 ºC and 32.75ºC. Formation differences between crystals A and C are of 15 to 20 mS/cm of surface and bottom EC, respectively; and of almost 5 ºC of brine temperature at both surface and bottom of the crystallizers. Moreover our results indicate that the difference between surface and bottom temperature during category A crystals formation was the lowest and the surface-bottom EC difference was negative, therefore opposite to the conditions prevailing during the formation of B and C crystal types.

Conclusions

The present study allowed to establish a relation between Fleur de sel crystals size and monitored weather conditions and brine physicochemical characteristics. Fleur de sel crystals were classified in 3 categories according to crystals surface which ranged from 7 to 24 mm^2.

The commercially most interesting crystals (category A) were harvested during the days of the highest incident solar radiation, prevailing southern winds, and the lowest wind speed and evaporation rate. Air temperature and air relative humidity did not show any relation to the different types of crystals. Maximum exposure to the southern of the salt crystallizers can be recommended to favour these conditions.

Brine physicochemical characteristics study showed that conditions favouring category A crystals were: high EC and temperature at the surface and bottom of the crystallizers.

Fleur de sel classification based on crystal size could be used for the commercial purpose. Currently, some Fleur de sel is commercialized as “salt flakes”, or large tabular crystals of unspecified size. However, this type of Fleur de sel is rarely produced entirely by natural solar evaporation and anthropogenic energy and different facilities need to be used to control temperature and time.

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