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Multielement analysis in beer cans using X-ray fluorescence

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ABSTRACT

In this study, the elemental concentrations of twenty-three beer cans of different brands and nationalities were determined using X-ray fluorescence technique. Beer cans from Brazil (20 beer cans), Germany, the United Kingdom and Colombia were analyzed, totalizing 15 different breweries. It was possible to calculate the concentration of 15 elements in the beer cans samples: Mg, Al, Si, S, Cl, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, Zr and Pb. The results showed that aluminum concentrations ranged from 94 % to 96 %. Other's elements can be considered trace elements, being the highest concentrations of them for Mg (approximately 2%) and Mn (approximately 1%). Some elements showed differences between cans from different nationalities, with Zn being much higher in samples from Brazil. The Zr element showed similarities between German and UK beer cans. In addition, the beer can sample from Colombia had lower concentrations of Cl and Ga. Tukey´s HSD test showed that some elements presents statistical differences in concentrations between different nationalities. This study shows that the values found are comparable to those found in the literature and that the concentrations of some trace elements (approximately 0.24 %) in beer cans may represent signs of impurities brought during the recycling process.

Keywords: **X-ray fluorescence, Beer can, Aluminum alloy.**

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1. INTRODUCTION

Beer consumption has become moderate to high in several countries, being a drink known worldwide [1]. Consumption of soda and beer in aluminum cans reaches 2 × 10^{11} cans every year [2, 3]. Trace metals in beer may originate from natural sources (soil, water, cereal, hops, and yeast) as well as from environmental contamination due to fertilizers, pesticides, industrial processing, and containers [1, 4]. The maximum concentrations of trace elements in beers are controlled by legislation, as these elements might be essential or toxic in the human body depending on their concentration [5]. For instance, lead is a highly toxic element that accumulates in biological systems and has a long half-life and Zinc is an essential element during the synthesis of proteins and energy metabolism [1, 6]. Thus, several metals, such as As, Pb and Cd, are subjected to careful control, not only related to beer production, but rather to the materials that come into contact (processing aids, additives, storage materials) [7].

The unique combinations of properties provided by aluminum and its alloys make aluminum one of the most versatile, economical, and attractive metallic materials for a broad range of uses—from soft, highly ductile wrapping foil to the most demanding engineering applications. Aluminum alloys are second only to steels in use as structural metals [8].

Aluminum fits can be used in food packaging and beverages due to its unique barrier and physical properties, effectively protecting food and drink against the quality-reducing effects of oxygen, light, moisture, micro-organisms, and unwanted aromas even in its thinnest form [3]. In 2007, approximately 30 percent of aluminum production in the country was destined for food industry and beverage packaging market, especially for beer cans [9].

Aluminum recycling has a number of key environmental and economic benefits. With these energy and cost savings in mind, many producers now have targets of increasing their usage of secondary materials. However, the accumulation of impurities in these recycled material streams may provide a significant compositional barrier to these goals. A growing number of studies and literature suggest that the accumulation of unwanted elements is a growing problem; for the case of aluminum, the list of problematic impurities is quite large, including but not limited to Si, Mg, Ni, Zn, Pb, Cr, Fe, Cu, V, and Mn [10].

Aluminum recycling involves three important steps. First, aluminum products that have been used and were collected, especially old soda and beer cans. Second, the cans are soaked in acid, so the designs and brand names are removed. Third, the cans are crushed and melted in a furnace. Once the cans have completely melted, the molten mixture is poured into molds, where it cools into solid aluminum bars called ingots. These bars are then transferred to another factory. There, they are melted again, and a special machine turns the liquid metal into blocks that are later used to make new aluminum items [11].

The removal of unwanted elements in the scrap stream is dictated by the energy considerations of the melting process. The melting temperature of many materials is lower than that of aluminum. For example, the melting temperature of lead (Pb) is 327°C and that of zinc (Zn) is 419°C while that of aluminum (Al) is 660°C adding impurities to the recycled material [10].

Although even with these negative aspects of impurities the increase in recycled metal becoming available is a positive trend for the environment, as secondary metal produced from recycled metal requires only about 2.8 kWh/kg of metal produced (about 7% of the energy needed to produce primary aluminum) while primary aluminum production requires about 45 kWh/kg of metal produced. It is to the industry and national advantage to maximize the amount of recycled metal, both from the energy savings and reduction of dependence upon overseas sources (now about 40% of U.S. consumption) and also from the ecological standpoint since recycling emits only about 4% as much CO_2 as primary production [12].

This study aimed to evaluate the elemental composition of twenty-three beer cans of different brands and nationalities through the X-ray fluorescence technique that could be useful as a reference for elements presented in aluminum beer cans. In addition, this work can work as a database on elemental composition to aid future research with beer cans.

2. MATERIALS AND METHODS

2.1. Samples

It was analyzed elemental concentrations of twenty-three beer cans. The beers are from 4 different nationalities, most of them Brazilian beers (20 beer cans), 1 beer can from Germany, 1 beer can from the United Kingdom and 1 beer can from Colombia. From a total of 20 Brazilian beers, it was analyzed 12 different national breweries.

XRF analysis of the aluminum can was performed using an internal side of the can cut out in a square with an area of 9 cm^2 (Figure 1). The analyzed areas were cleaned with 70% alcohol to remove the remaining beer and impurities in them. The internal side of the can (sample) was analyzed so that the paint or film used on the external side of the can would not influence the XRF measurement. The preparation sample was done so that the inside of the can was flat, without residues and the beam irradiated the entire sample. The thickness of Brazilian beer cans was (118 ± 31) µm while other nationalities were (106 ± 4) µm. Samples were analyzed in triplicate.

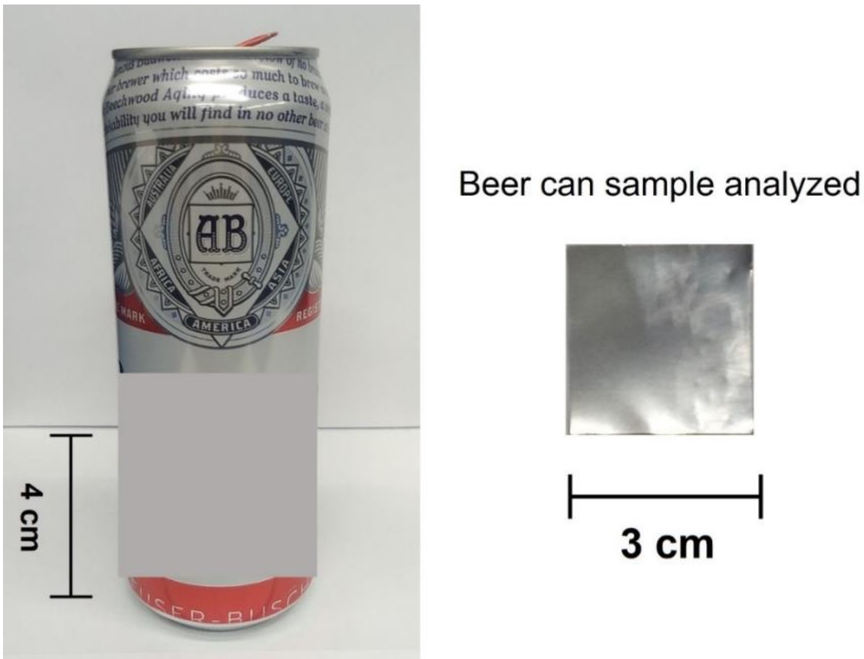

Figure 1: *Example of an analyzed beer can.*

2.2. XRF analysis

EDXRF analysis of the beer cans was performed using the commercial equipment Epsilon 1 (Malvern Panalytical) with silver anode (Ag) and SDD detector (Silicon Drift Detector) (Energy resolution < 135 eV for Mn-Kα). Analysis was performed in two different experimental conditions for the same sample. First experimental condition was used for better excitation of low Z elements

(Mg to Ca K-lines), using 10 kV, 500 µA and an acquisition time of 180 s. Whereas, the second experimental condition was used to excitation high Z elements (V to Zr K-lines and Pb L-lines), using 50 kV, 100 µA, 300 s and a Cu filter (500 µm). Quantitative analyses were carried out by the own software of Epsilon 1 (Malvern Panalytical). Samples were analyzed in triplicate.

For the purpose of checking the precision and the accuracy of the EDXRF system, we carried out the elemental analysis of a Reference Material: DIN1725. The reference material samples were prepared under the same experimental conditions as beer cans samples. All experiment was performed under normal atmospheric conditions at room temperature. The EDXRF spectra were processed using the own software of Epsilon 1 (Malvern Panalytical) and reference sample was prepared to ensure that it was flat and was fully irradiated. The sample was analyzed in triplicate.

A frequently used pairwise comparison technique was developed by Tukey under the name of the honestly significant difference (HSD) test. The main idea of the HSD is to compute the honestly significant difference (i.e., the HSD) between two averages using statistical distribution. This distribution gives the exact sampling distribution of the largest difference between a set of averages originating from the same population. All pairwise differences are evaluated using the same sampling distribution used for the largest difference [13]. Multiple comparisons of averages allow to examine which means are different and to estimate by how much they are different [14]. In this work, Tukey´s HSD test was performed with a confidence interval of 95%. Statistical analysis was performed using SPSS 22.0 (IBM, Armonk, NY, USA).

3. RESULTS AND DISCUSSION

Analytical quality control was ensured by analysis of a certified reference material DIN1725. Table 1 shows the results obtained for reference material: the average concentrations, standard deviation, relative error and relative standard error (RSD). The relative errors ranged from 2 % (Al) to 20 % (Mg). In addition, relative standard deviations (RSD) were distributed in a range of 0.04 % (Al) to 7% (Si). Table 1 shows a comparison between the results obtained in this work using EDXRF technique and the certified values. These results show that the EDXRF system presented good accuracy (except for Mg 20% with 20% of relative error). The relative error for Mg can be associated to the low concentration in the sample and low Z element. In general, the results obtained for DIN1725 were in agreement with the comparative certified values that were previously obtained, indicating that the method provides comparable results.

Table 1: Comparison between values obtained and certified values
(Mean value ± standard deviation and relative standard deviation)

Elements	**DIN1725**[a]	**Epsilon 1**	**Relative Error (%)**	**RSD (%)**
Mg (%)	0.05	0.060 ± 0.002	20	4
Si (%)	0.25	0.21 ± 0.01	16	7
Al (%)	99.50	97.97 ± 0.04	2	0.04
Fe (%)	0.40	0.350 ± 0.005	13	2
Cu (%)	0.05	0.040 ± 0.001	11	2
Zn (%)	0.07	0.060 ± 0.001	9	2

a. Certified sample DIN1725.
b. Epsilon 1 (Malvern Panalytical)

Figure 2 shows the X-ray spectra of a Brazilian beer can sample (low Z and high Z experimental conditions). It was possible to identify and determine the concentrations of 15 elements (Mg, Al, Si, S, Cl, V, Cr, Mn, Fe, Ni, Cu, Zn, Ga, Zr and Pb) for all 4 nationalities. The element Argon (Ar) element is present in the spectrum due to the fact that the measurements were not carried out in vacuum, the Ar concentration is about 1.0% in a standard atmosphere.

Table 2 shows the average, minimum and maximum concentrations found in samples of beer cans. Twenty-three aluminum samples were analyzed and in all of them, Al was the major element (approximately 95 %). The aluminum alloy 3104, used for beverage can production, can mainly consist of 95.6 – 98.2 % Al, 0.8 – 1.4 % Mn and 0.8 – 1.3 % Mg [15].

Table 3 shows the results of Tukey's HSD test comparing the elemental concentrations of beer cans of different nationalities with a significance level of 5%.

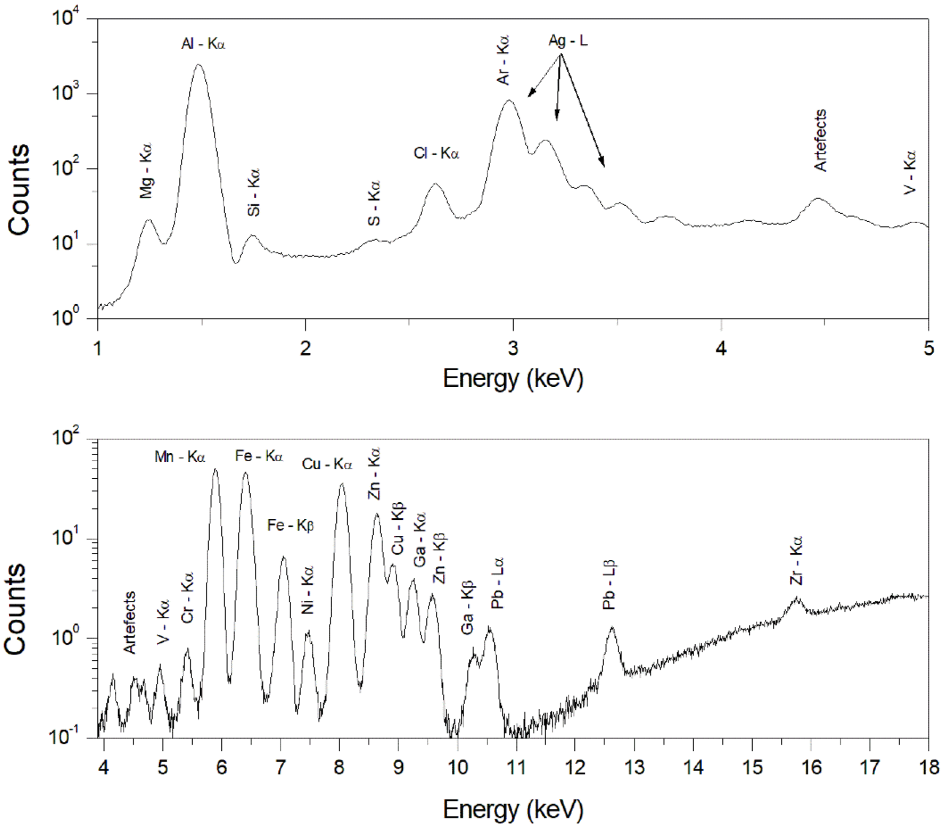

Figure 2: ED*XRF spectra of a beer can sample (low Z and high Z experimental conditions)*

During recycling other elements such as iron (Fe) may be expected to gradually increase with the number of recycling processes and may require special attention; Magnesium (Mg), Nickel (Ni) and Vanadium (V) are other examples. Table 4 shows the values defined for weight concentration (%) of aluminum alloys 3104 and 3105, which are normally used for the body of beverage cans [16].

In beer cans, another 5 elements added to Al contribute more than 99% of the concentration, Mg, Si, Mn, Fe and Cu. Therefore, the other elements found in beer cans can be considered trace elements (S, Cl, V, Cr, Ni, Zn, Ga, Zr and Pb). The majority elements showed concentrations below the values of aluminum alloy 3104 as used as reference, except for Mg and Fe.

The average concentrations of Mg are above the reference values (3104) for cans of different nationalities, approximately 2 times higher. The analyze of Mg that is a low Z element using XRF technique was impaired due the experiment was carried out at room temperature without vacuum.

Table 2: Elemental concentrations of the beer can
(Mean value ± standard deviation and minimum to maximum concentrations)

Elements	**Brazil**[a]	**Germany**	**United Kingdom (UK)**	**Colombia**
Mg (%)	2.4 ± 0.2[b]	2.2 ± 0.1	2.6 ± 0.3	2.5 ± 0.2
	1.7 – 2.7[c]	2.1 – 2.4	2.2 – 2.9	2.4 – 2.7
Al (%)	95 ± 1	95.1 ± 0.2	94.9 ± 0.3	95.1 ± 0.7
	94.7 – 96.1	94.9 – 95.3	94.6 – 95.2	94.4 – 95.6
Si (%)	0.46 ± 0.03	0.53 ± 0.01	0.49 ± 0.01	0.41 ± 0.01
	0.4 – 0.5	0.52 – 0.54	0.49 – 0.50	0.41 – 0.42
S (µg.g^{-1})	301 ± 183	124 ± 5	157 ± 8	155 ± 27
	101 – 645	120 – 127	149 – 165	124 – 174
Cl (µg.g^{-1})	1243 ± 292	1427 ± 257	1014 ± 347	804 ± 155
	829 – 1860	1130 – 1580	763 – 1410	711 – 984
V (µg.g^{-1})	161 ± 17	133 ± 11	157 ± 4	126 ± 5
	137 – 193	121 – 142	152 – 161	120 – 130
Cr (µg.g^{-1})	162 ± 17	183 ± 8	186 ± 9	155 ± 8
	120 – 192	174 – 190	181 – 197	149 – 165
Mn (%)	1.0 ± 0.1	1.1 ± 0.1	0.99 ± 0.01	0.86 ± 0.02
	0.8 – 1.1	1.0 – 1.1	0.98 – 0.99	0.84 – 0.88
Fe (%)	0.51 ± 0.05	0.66 ± 0.04	0.64 ± 0.01	0.42 ± 0.01
	0.4 – 0.6	0.61 – 0.69	0.64 – 0.65	0.42 – 0.44
Ni (µg.g^{-1})	71 ± 7	75 ± 5	79 ± 2	62 ± 2
	58 – 80	68 – 79	78 – 82	60 – 63
Cu (%)	0.17 ± 0.02	0.19 ± 0.01	0.16 ± 0.01	0.16 ± 0.01
	0.14 – 0.20	0.18 – 0.20	0.16 – 0.17	0.16 – 0.17
Zn (µg.g^{-1})	601 ± 77	342 ± 23	466 ± 10	480 ± 10
	480 – 760	317 – 361	460 – 477	474 – 491
Ga (µg.g^{-1})	94 ± 9	103 ± 7	92 ± 2	77 ± 1
	79 – 110	95 – 108	90 – 94	76 – 78
Zr (µg.g^{-1})	7 ± 1	21 ± 3	16.6 ± 0.6	5.3 ± 0.2
	6 – 12	18 – 23	16 – 17	5.1 – 5.5
Pb (µg.g^{-1})	37 ± 5	18 ± 1	22.9 ± 0.7	31.5 ± 0.4
	28 – 48	17 – 19	22 – 24	31 – 32

a. Values obtained from 20 samples of beer cans.
b. Mean value ± standard deviation.
c. Minimum – maximum values.

Table 3: Tukey´s HSD test

Elements	Location (i)	Location (j)			
		Brazil	Germany	United Kingdom (UK)	Colombia
Mg	Brazil	1.000			
	Germany	0.653	1.000		
	United Kingdom (UK)	0.340	0.155	1.000	
	Colombia	0.720	0.351	0.958	1.000
Al	Brazil	1.000			
	Germany	0.972	1.000		
	United Kingdom (UK)	0.402	0.814	1.000	
	Colombia	0.992	0.999	0.754	1.000
Si	Brazil	1.000			
	Germany	0.001*			
	United Kingdom (UK)	0.147	0.272	1.000	
	Colombia	0.005*	<< 0.05*	0.001*	1.000
S	Brazil	1.000			
	Germany	0.319	1.000		
	United Kingdom (UK)	0.496	0.994	1.000	
	Colombia	0.481	0.995	1.000	1.000
Cl	Brazil	1.000			
	Germany	0.733	1.000		
	United Kingdom (UK)	0.579	0.312	1.000	
	Colombia	0.093	0.061	0.806	1.000
V	Brazil	1.000			
	Germany	0.030*	1.000		
	United Kingdom (UK)	0.968	0.241	1.000	
	Colombia	0.005*	0.939	0.085	1.000
Cr	Brazil	1.000			
	Germany	0.119	1.000		
	United Kingdom (UK)	0.063	0.995	1.000	
	Colombia	0.892	0.123	0.076	1.000
Mn	Brazil	1.000			
	Germany	0.547	1.000		
	United Kingdom (UK)	0.986	0.584	1.000	
	Colombia	0.015*	0.009*	0.137	1.000

Continuation

Elements	Location (i)	Location (j)			
		Brazil	Germany	United Kingdom (UK)	Colombia
Fe	Brazil	1.000			
	Germany	<< 0.05*	1.000		
	United Kingdom (UK)	0.000*	0.971	1.000	
	Colombia	0.019*	<< 0.05*	<< 0.05*	1.000
Ni	Brazil	1.000			
	Germany	0.742	1.000		
	United Kingdom (UK)	0.123	0.758	1.000	
	Colombia	0.133	0.085	0.010*	1.000
Cu	Brazil	1.000			
	Germany	0.062	1.000		
	United Kingdom (UK)	0.865	0.068	1.000	
	Colombia	0.882	0.073	1.000	1.000
Zn	Brazil	1.000			
	Germany	<< 0.05*	1.000		
	United Kingdom (UK)	0.017*	0.136	1.000	
	Colombia	0.037*	0.082	0.994	1.000
Ga	Brazil	1.000			
	Germany	0.327	1.000		
	United Kingdom (UK)	0.972	0.370	1.000	
	Colombia	0.013*	0.004*	0.144	1.000
Zr	Brazil	1.000			
	Germany	<< 0.05*	1.000		
	United Kingdom (UK)	<< 0.05*	0.011*	1.000	
	Colombia	0.137	<< 0.05*	<< 0.05*	1.000
Pb	Brazil	1.000			
	Germany	<< 0.05*	1.000		
	United Kingdom (UK)	<< 0.05*	0.630	1.000	
	Colombia	0.234	0.008*	0.117	1.000

* p = 0.05

Table 4: Defined values for 3104 and 3105 aluminum alloys (Values in weigh %)

Alloy	**Cu**	**Fe**	**Mg**	**Mn**	**Si**	**Zn**	**Others**
3104 (%)	0.80*	0.60	0.8 - 1.3	0.8-1.4	0.60*	0.25*	0.15*
3105 (%)	0.30*	0.70*	0.20 - 0.80	0.30 - 0.80	0.60*	0.40*	0.15*

* This value represents the maximum allowed value.

The average concentration of Fe for German and British beer cans is above the maximum allowed limits if we consider alloy 3104 as a reference. Tukey's test showed significant differences between these two beer cans and the Colombian beer can ($p << 0.05$ for both cans) and the average concentration of the Brazilian cans ($p << 0.05$ for both beers). On the other hand, the Fe average concentration in Brazilian beer cans also showed a significant difference in relation to the Colombian beer cans ($p = 0.019$).

The concentration of Si showed significant differences between cans of different nationalities. However, all average values were below the reference value. The Colombian beer can obtained the lowest average concentration, showing a statistical difference from the cans of other nationalities. The German beer can had the highest average concentration, showing statistical differences from Brazilian beer cans ($p = 0.016$).

The Mn concentration showed significant differences between Colombian beer cans and Brazilian and German beer cans ($p = 0.015$ and $p = 0.009$, respectively). However, all average values were below the reference value (3104).

Copper concentrations for beer cans of different nationalities are below the reference values (3104) and did not show statistical differences. On the other hand, Zn does not appear as a major element in this study. The reference values (3104) indicate that Zn can have concentration of up to 0.25 %, even though, in this study the maximum mean concentration of Zn was 608 ± 80 $ug.g^{-1}$ (or approximately 0.06 %) that is well below the established maximum limit.

The other elements found in beer cans can be considered trace elements, S, Cl, V, Cr, Ni, Zn, Ga, Zr and Pb. These elements together contribute approximately 0.225 – 0.243 % of the concentration. Some of these elements showed statistical differences in concentrations between the different nationalities, but all of them with a concentration at ppm level. This variation in the concentration of these trace elements supports the hypothesis that the recycling process may lead to higher levels of some elements than expected. This analysis shows that the values found are comparable to those found in the literature and that the concentrations of the elements in the beer cans may represent evidence of impurities brought during the recycling process.

4. CONCLUSION

Through the EDXRF technique, it was possible to calculate the concentration of 15 elements in the beer cans samples. A majority concentration of Aluminum of around 95% was obtained. All the other elements can be considered trace elements, being the highest concentrations of them for Mg (approximately 2%) and Mn (approximately 1%). Some elements showed differences in concentrations between cans from different nationalities, with Zn being much higher in samples from Brazil. The Zr element showed similarities between German and UK beer cans. In addition, the beer can sample from Colombian had lower concentrations of Cl and Ga. This study also showed that there are statistical differences of beer cans from different nationalities.

There are many advantages to recycling beer cans, such as energy, economic and environmental benefits. However, during this process there is also the possibility of propagation of impurities such as Pb, S and Ga in values not tabulated and quantified. A solution to reduce elements called impurities would be the separation before the melting process.

The presence of some elements in the human body can have toxic effects depending on their concentration. The presence of undesirable elements in beer liquid can be a consequence of the aluminum can and could influence the concentration of some elements in the beer. Therefore, this work has as a future perspective the analysis of the liquid of these beers using the technique of Total Reflection X-Ray Fluorescence (TXRF) to investigate and compare the results obtained from the cans with the beer liquid, and thus identify possible transitions from one to the other.

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