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Mass Transfer of Dichloromethane from EU Retail Roast and Ground Decaffeinated Coffee into Prepared Beverages †

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Abstract: Dichloromethane (DCM) is extensively used around the globe in various applications, such as in closed industrial installations for food processing or pharmaceutical extractions (vitamins, antibiotics, etc.). In the coffee sector, it is used as an extraction agent for the decaffeination process of green coffee beans. Due to its low boiling point, ranging at approx. 40 °C, DCM can be easily removed subsequent to caffeine extraction by applying state-of-the-art solvent stripping processes. The intention of this study is to assess how much DCM, if any, is present in decaffeinated coffee packages as sold to the consumer, as well as how much of the extraction solvent residue is transferred into the finally prepared, consumable coffee beverage. This study sets out to highlight DCM contents of decaf coffees, directly taken from six EU countries' supermarket shelves. In addition, DCM mass transfer rates from roasted coffee matrices into the corresponding, variously prepared beverages (drip percolated coffee, French press) are determined. All analyses were performed applying a Headspace-GC-MS technique. All presented data demonstrate that DCM residues in the 34 coffee samples analysed have contents well below the DCM maximum residue limits for roasted coffee both in the European Union (2 mg/kg) and the USA (10 mg/kg), with an average of 0.127 mg/kg, median value of 0.059 mg/kg and P95 of 0.444 mg/kg. Furthermore, this study shows that DCM mass transfer rates from the coffee matrices into the corresponding beverages have, for drip coffee, an average of 24.7% and median of 26.8%, and for French press, an average mass transfer of 41.9% and median of 43.1%.

Keywords: dichloromethane; decaffeination; roasted coffee; drip coffee; French press

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1. Introduction

The intention of this study is to review how much dichloromethane (DCM) is present in decaffeinated coffee consumer packages when DCM is used as an extraction solvent and how much is actually transferred to beverages consumed.

For the decaffeination process of green coffee, dichloromethane is used around the globe extensively as an extracting agent, where the solvent is in direct contact with the green coffee beans. Due to its low boiling point, dichloromethane can be easily removed after caffeine extraction by vaporization. To prevent any risk to human health, maximum residue levels of dichloromethane in roasted coffee were set in Europe and the US.

This study analyses the DCM content of coffees provided to the consumer and compares the transfer from roasted coffee into two differently brewed beverages, drip coffee and French press, respectively.

2. Materials and Methods

2.1. Coffee Samples

The samples to be analysed for dichloromethane were 34 different commercially available roasted decaffeinated coffee products. In total, 28 samples were decaffeinated, roasted and ground coffee; three samples were decaffeinated and roasted whole coffee beans; and three samples decaffeinated, roasted and ground coffee in capsules.

All 34 samples were brewed as drip coffee and in a French press. Samples of all preparations were analysed on dichloromethane directly after beverage preparation.

For the analysis of dichloromethane, the following equipment were used: headspace gas chromatograph 7890 with MSD 5977 A supplied by Agilent Technologies Inc., Santa Clara, CA, USA, coupled with a MPS 2 autosampler and cold injection system KAS 4 with Carbotrap liner, supplied by Gerstel GmbH & Co.KG, Mülheim an der Ruhr, Germany. The chromatographic column was J&W PoraPlot Q 25 m × 0.32 mm ID, 0.45 µm film, supplied by Agilent Technologies Inc., Santa Clara, CA, USA and the carrier gas was helium.

In total, 2.5 g coffee powder or 10 mL brew sample was transferred into a 20 mL headspace vial. Coffee powder was covered with 10 mL purified water. Incubation time for the vial was 10 min at 80 °C. Injection volume was 2.0 mL gas sample in solvent venting mode. Quantification was carried out using an external 6-point matrix calibration with dichloromethane standard (LGC). DCM was identified and quantified at m/z 84 and 86, respectively, in ESI-positive SIM mode.

2.2. Coffee Beverage Preparation

The three samples of roasted whole beans were ground with a Mahlkoenig®EK43S coffee mill supplied by Hemro International AG, Zurich, Switzerland, with typical medium coarseness required for the subsequent extraction.

For the brewing preparation of the three samples of capsules, the capsules were opened and the content of ten capsules of each sample was mixed and used for the extraction.

2.3. Preparation of Drip Coffee

Filter drip coffee preparation was performed manually with a V60 dripper supplied by Hario Europe, Amstelveen, The Netherlands. The V60 coffee filter paper was rinsed with hot water, and 20 g ground (medium coarseness) coffee powder was weighed into the filter. The ground coffee was moistened with 90–95 °C hot water until it was completely covered and simmered for about 30 s. In circular motions, the remaining total 300 g hot water was poured on top. If necessary, the filter was stirred briefly. After the extract had completely drained through the coffee bed, the beverage sample flask was directly closed and analysed.

2.4. Preparation of Coffee in the French Press

For the preparation of coffee samples in the French press, 20 g of coffee powder was directly weighed into the French press and 300 g of hot water (90–95 °C) was poured over. The extract was stirred briefly, and the lid was lightly pressed on the surface so that the coffee powder was completely covered with water. After approx. three minutes, the lid was pressed down slowly and evenly. The freshly prepared coffee was directly filled in a sample flask and analysed.

3. Results

3.1. DCM in Original R&G Coffee Samples

The 34 original R&G coffee samples have concentrations ranging from 5.7 to 816.6 µg/kg (Figure 1), with an average of 127 µg/kg, median value of 59.5 µg/kg and P95 of 443.6 µg/kg.

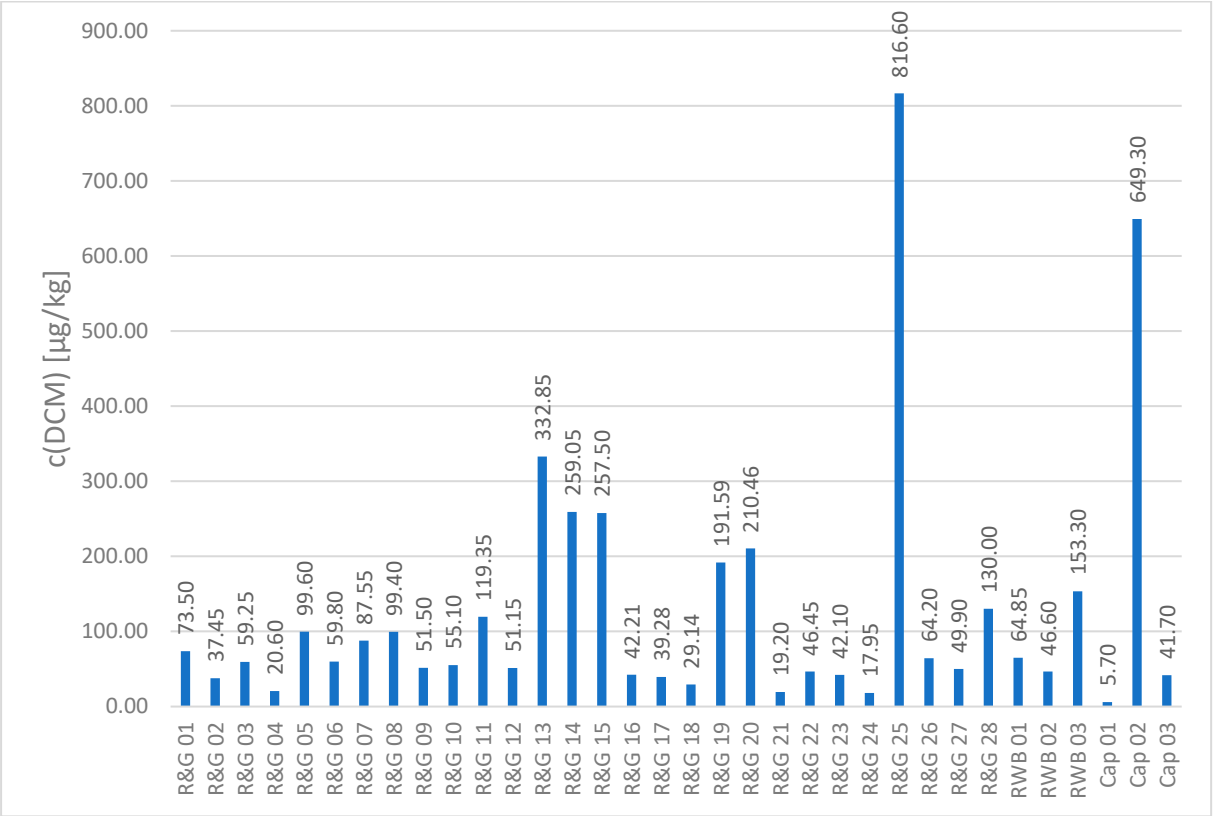

Figure 1. DCM concentration in original R&G coffee samples in µg/kg.

3.2. DCM Content per Portion and Transfer Rate in the Beverage

Based on the above results, the DCM content per portion in original R&G coffee samples (20 g) was calculated and the results were compared with the content per portion in drip coffee (0.3 L) and French press (0.3 L), as resumed in Table 1; then, the transfer rates were calculated and reported both in Table 1.

Table 1. DCM content per portion original R&G coffee sample (20 g), drip coffee (0.3 L) and French press (0.3 L) and transfer rate in the beverage.

Sample	Sample Type	Original R&G Coffee	Drip Coffee		French Press	
		m(DCM) [µg] in 20 g Portion	m(DCM) [µg] in 0.3 L Portion	DCM Transfer [%]	m(DCM) [µg] in 0.3 L Portion	DCM Transfer [%]
R&G 01	R&G coffee, decaf	1.47	0.315	21.4	0.519	35.3
R&G 02	R&G coffee, decaf	0.75	0.094	12.5	0.191	25.5
R&G 03	R&G coffee, decaf	1.19	0.273	23.1	0.336	28.3
R&G 04	R&G coffee, decaf	0.41	<0.075	0.0	<0.075	0.0
R&G 05	R&G coffee, decaf	1.99	0.577	29.0	0.845	42.4
R&G 06	R&G coffee, 50% caffeine	1.20	0.214	17.9	0.344	28.8
R&G 07	R&G coffee, decaf	1.75	0.464	26.5	0.626	35.8
R&G 08	R&G coffee, decaf	1.99	0.659	33.1	0.906	45.6
R&G 09	R&G coffee, decaf	1.03	0.238	23.1	0.472	45.8
R&G 10	R&G coffee, decaf	1.10	0.294	26.7	0.448	40.7

Table 1. *Cont.*

Sample	Sample Type	Original R&G Coffee	Drip Coffee		French Press	
		m(DCM) [µg]	m(DCM) [µg]	DCM Transfer	m(DCM) [µg]	DCM Transfer
		in 20 g Portion	in 0.3 L Portion	[%]	in 0.3 L Portion	[%]
R&G 11	R&G coffee, decaf	2.39	0.714	29.9	0.956	40.1
R&G 12	R&G coffee, decaf	1.02	0.235	22.9	0.398	38.9
R&G 13	R&G coffee, decaf	6.66	2.024	30.4	4.985	74.9
R&G 14	R&G coffee, decaf	5.18	3.080	59.4	5.246	101.2
R&G 15	R&G coffee, decaf	5.15	2.213	43.0	4.011	77.9
R&G 16	R&G coffee, decaf	0.84	0.230	27.3	0.387	45.8
R&G 17	R&G coffee, decaf	0.79	0.099	12.6	0.252	32.1
R&G 18	R&G coffee, decaf	0.58	<0.075	0.0	0.102	17.5
R&G 19	R&G coffee, decaf	3.83	1.029	26.9	1.880	49.1
R&G 20	R&G coffee, decaf	4.21	1.008	23.9	2.103	50.0
R&G 21	R&G coffee, decaf	0.38	<0.075	0.0	0.125	32.4
R&G 22	R&G coffee, decaf	0.93	0.341	36.7	0.560	60.2
R&G 23	R&G coffee, decaf	0.84	0.234	27.8	0.401	47.6
R&G 24	R&G coffee, decaf	0.36	<0.075	0.0	<0.075	0.0
R&G 25	R&G coffee, decaf	16.33	6.345	38.9	9.053	55.4
R&G 26	R&G coffee, decaf	1.28	0.378	29.4	0.600	46.7
R&G 27	R&G coffee, decaf	1.00	0.362	36.3	0.438	43.9
R&G 28	R&G coffee, decaf	2.60	1.131	43.5	1.296	49.8
RWB 01	Roasted, whole beans, decaf	1.30	0.307	23.7	0.449	34.6
RWB 02	Roasted, whole beans, decaf	0.93	0.294	31.6	0.496	53.2
RWB 03	Roasted, whole beans, decaf	3.07	1.111	36.2	1.743	56.9
Cap 01	R&G coffee capsules, decaf	0.11	<0.075	0.0	<0.075	0.0
Cap 02	R&G coffee capsules, decaf	12.99	6.050	46.6	11.454	88.2
Cap 03	R&G coffee capsules, decaf	0.83	<0.075	0.0	<0.075	0.0
	Average	2.54	1.08	24.71	1.72	41.90
	Median	1.19	0.37	26.79	0.54	43.14
	P95	8.87	5.01	44.59	7.34	81.50

4. Conclusions

The data presented show that DCM residues in the 34 samples analysed, directly taken from six EU countries' supermarket shelves, have contents well below the DCM maximum residue limit for roasted coffee in the EU (2 mg/kg) or USA (10 mg/kg), with an average of 0.127 mg/kg, median value of 0.059 mg/kg and P95 of 0.444 mg/kg.

Furthermore, this study shows that the DCM mass transfer rates from the coffee matrices into the corresponding beverages have an average of 24.7% (median of 26.8%) for drip coffee, and average mass transfer of 41.9% (median of 43.1%) for French press,

demonstrating that the brewing process contributes to a further reduction in the DCM content in the beverage.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/ICC2024-18166/s1, Presentation file.

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