

Cobalt release of oxygen scavenger added multilayered PET bottles in various domestic solvents

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Abstract. Cobalt catalyzed oxygen scavenger added multilayered PET bottles were inspected in our work. X-ray fluorescent spectrometry (XRF) technique was used to select the investigated bottles. Inductive coupled plasma mass spectrometry (ICP-MS) was applied to determine the cobalt content of different domestic solvents after extraction. Extraction experiments were separated into 48 hour extractions, Lakanen-Erviö extraction, and extraction kinetic experiments. The fruit brandy and the orange juice contained in the bottles were the two most successful agents dissolving cobalt without chemical reactions. 1 M NaOH was the agent dissolving the highest quantity of cobalt as a consequence of the hydrolysis of PET. Continuous cobalt dissolving over time could be experienced when NaOH agent penetrated into the grains of flakes. Reuse of active multilayered bottles for domestic solvents did not show harmful cobalt concentration, but the ground and oxygen scavenger added multilayered PET bottles showed cobalt concentration over the limit value.

1. Introduction

Nowadays, multilayered PET bottles are widely used for juice packaging [1]. These types of bottles, produced by three or five layered preform molding technology, give favorable packaging features. The layers made of different linear polyester compounds show passive barrier [2], and active blocking function. With this up-to-date technology, five layered PET bottles were made so that the outer and the inner layers were made of virgin PET raw materials, but the three layers situated in the middle were made of different blended polyesters. The middle layer, as an active blocking layer, serves as an oxygen scavenger layer. The function of the scavenger layer is that oxygen diffusion into the bottle is hindered by additional chemicals, e.g. cobalt salts. Due to this favourable oxygen scavenger feature, a wide range of oxygen sensitive products such as tomato-based products, UHT milk, fruit juice are often bottled into active oxygen scavenger layered PET bottles. Some difficulties arise during the recycling process operations of multilayered PET bottles. Currently, blended multilayered PET bottles and mono layered bottles undergo the same recycling procedure. Large amounts of bottles or regranulates are transported from Europe to countries in the Far East as raw material for the textile industry. A small number of ground or compressed bottles finish their life cycle in communal waste-piles, oceans, or living waters. Due to their practical form and strength, the PET bottle recycling process begins in the home when acidic, basic or alcoholic liquids are used to refill the bottles. In recycling sites the used PET bottles are selected by their colour, but they are ground, mechanically washed in common soda solvents, and the final steps are for them to be dried, re-granulated, and



recrystallized. Tons of ground PET flakes, as ready to use recycling, are on the market. Several technical publications explain the short lifetime and the appropriate process technology for multilayered PET bottles [1]. Due to the new and profitable feature of active and passive oxygen scavenger layer technology [2], only a few scientific publications are available to explain the chemical components and the way these special polyester blends operate [3,10]. The present publication aims to describe the cobalt release of the oxygen scavenger [4,5] added multilayered PET bottles exclusively in domestic solvents in order to verify the possibility of safe application following commercial use. Another aim is to define the quantity of extractable cobalt ions in order to assess the impacts of these extracted amounts on the environment. In the course of our series of experiments, cobalt catalyzed oxygen scavenger added PET bottles separated by X-ray fluorescent spectrometry (XRF) were tested by inductive coupled plasma mass spectrometry (ICP-MS). We determined the cobalt content of different domestic solvents. The extraction experiments were divided into 48-hour extractions, Lakanen-Erviö extraction, and extraction kinetic experiments.

2. The function and operational mechanism of active scavenger in PET bottles

The active scavenger layer applied in multilayer bottles consumes oxygen that permeates through bottles thereby hindering the adverse effects caused by oxygen. This system stays active until there is a sufficient amount of additive material in order to maintain an oxidation reaction. The oxygen scavenger system is a multicomponent system and the reaction itself is a radical reaction, i.e. oxidative degradation, and follows three steps analogous to free-radical polymerization reactions: initiation, propagation, termination. In the initiation step a free-radical is formed by a free-radical donor. Usually this is conducted by the addition of peroxides, hydroperoxides or α , α -azobisisobutyronitrile. However, it is not essential to use such initiators. The autoxidation reaction may be initiated by the presence of light or heat as well. In the propagation step the addition of oxygen to alkyl free-radicals and abstraction of hydrogen from the polymer-chain can be observed. The polymer should be unsaturated and oxidizable and should have a so-called oxygen scavenging moiety, e.g. olefin functional group. These functional groups form peroxy ($R-O-O\cdot$) and alkoxy ($R-O\cdot$) free-radicals in combination with oxygen. Then peroxides ($R-O-O-R$) and hydroperoxides ($R-O-OH$) are formed, and finally they decompose into carbonyl groups ($>C=O$). The saturation of such an active system takes place when these oxygen scavenging moieties are depleted. Oxidizable polymers are polyamides or 1,4-polybutadiene. Intramolecular and intermolecular oxidation can be assumed in the reaction. Transition metal ions have an important role in the redox-decomposition of alkyl hydroperoxides. Most often cobalt and magnesium are used, but silver, cerium, iron and copper are also suitable for such purposes. In the case of polymers, the presence of a transition metal complex leads to decomposition, and, therefore, the production rate of alkoxy and alkylperoxy radicals will be increased. Thus the reaction is catalyzed in the initiation and in the propagation steps. The presumable mechanism of the catalytic reaction is an oxidation-reduction catalytic cycle in which the valence state of metal ions changes back and forth. The most effective transition metal complexes are cobalt carboxylates. Such metal complexes are cobalt octoate, cobalt stearate and cobalt neodecanoate. There are several active oxygen scavenger systems available on the market: AmosorbTM DFC [6], PolyshieldTM, AegisTM OX, OxbarTM and BindoxTM. AmosorbTM DFC is a PET masterbatch which can be added into any bottle grade PET during manufacture and the mixture thus obtained can be used to create an active scavenger layer.

3. Characteristics of cobalt and its salts, physiological, environmental impacts, and environmental limits

In nature, cobalt can be found in small quantities. It is present in the soil, natural bodies of water, plants, grain, and seeds, and it can be also found in animals. The concentration of cobalt in soil varies widely. It is usually about 1 to 40 mg/kg. Soils containing less than about 3 mg/kg of cobalt are considered cobalt-deficient. Plants growing in them do not have a sufficient amount of cobalt, and, therefore, these plants are not suitable to use as forage. In surface waters and ground waters cobalt can

be found in the form of ions normally in the range of ppb. Due to certain industrial activities, the concentration of cobalt may increase in natural bodies of water, the air and the soil. Therefore, in such areas, authorities may require a sampling of these media and analysis of these samples at given times or time intervals. In the connection with human health, cobalt has beneficial and harmful effects. Cyanocobalamin, or vitamin B12, is an essential compound of cobalt needed to maintain human and also animal health. Vitamin B12, together with the other B7 vitamins, helps the body convert carbohydrates into glucose [9]. Vitamin B12 plays an important role in sustaining healthy nerve cells, controlling blood levels of homocysteine, aiding the production of RNA, DNA, and also red blood cells [7]. For humans, good dietary sources of vitamin B12 are meat and dairy products, eggs and fish. The Recommended Dietary Allowance of vitamin B12 for an adult of average body weight is 2,4 µg/day, which means 0,1 µg of cobalt per day. Cobalt has been found in most tissues of the human body, but the highest concentrations are located in the liver [8].

In recent years increasing attention has been paid to the quality and the extent of contamination, including monitoring the quantitative changes, of soil as a geological medium and also of natural waters. In Hungary, regulation No 6/2009. (IV. 14.) KvVM-EüM-FVM is currently in force with respect to the sampling and administration of analytical measurements as well as the limit values of different contaminants in the geological media and ground waters. According to this regulation, the relevant limit values for cobalt are as follows: in geological media: 30 mg/kg dry matter, hazard classification K2; in ground waters: 20 ppb, hazard classification K2 [11].

Cobalt is an off-white ferromagnetic metal. Its reaction with non-metallic elements leads to +2 and +3 oxidation states, but there are known examples where 0, +1 or +4 oxidation states occur. Its reaction with carbon monoxide at 200 bar and at 150°C results in dicobalt octacarbonyl $[\text{Co}_2(\text{CO})_8]$. The color of aqueous solutions of Co(II) salts is typically pink due to the cobalt(II) hexahydrate $[\text{Co}(\text{H}_2\text{O})_6]^{+2}$ ions, while the color of anhydrous cobalt salts are typically blue (Co^{+2}).

4. Experimental materials and methods

Knowing that the bottle production line middle layer had an active scavenger layer made by an AmosorbTM additive, bottles of high-fiber content orange juice having three-layers were purchased from consumer markets. The matrix of AmosorbTM was polyethylene terephthalate, the active component was 1,4- polybutadiene, and the free radical formation initiator was some kind of cobalt carboxylate. To increase the efficiency of the extraction and to copy the first step of the recycling process, we chose a grinding sample preparation. A Piovani's RSP 15/15 B2RX granulator (serial number: 110000002898) was used for grinding 40 pieces of one litre bottles of a well-known brand of 100% high-fiber orange juice. Before grinding we randomly selected 5 bottles to determine their average weight and cobalt concentration. The cobalt concentration was measured by a ED-XRF spectrometer. Based on the ED-XRF measurements, the Co concentration showed homogeneity. A sieve analysis was used to validate the grinding by using 4.00 mm, 3.15 mm, 2.00 mm, 1.00 mm, 0.5mm, and 0.315 mm sieves. From each sieve fraction a 0.5 gr sample was taken in order to determine the average cobalt concentration. The average cobalt concentration of ground bottles was 36.805 mg/kg. This value was higher than the cobalt limit value for geological media (30 mg/kg dry matter). The average cobalt content for a one litre bottle was calculated and is 1.014mg/bottle. The domestic solvents which were selected were ones that can be found in households and which may possibly come into contact with mono or multilayer active blocker bottles (detergents, fruit brandy, etc.). In some cases, we use more powerful solvents in our households than those which may be found in a waste landfill. Such solvents, 1 M NaOH, HCl, ammonia, etc., were used in our experiments. The parameters of the 48-hour extraction experiments and the used solvents are shown in Table 1. The density of the traditional fruit brandy and ethanol volume percentage was measured in the Food Laboratory Department of Bálint Analytics Ltd. During the extraction and sample preparation the weight was measured by a digital mass balance. Flakes were homogenized by a strong shake of the powder in the container bag. The various solvents were dosed by a graduated cylinder. A heated magnetic stirrer (with 12 blades) was applied for the extraction preparations. The experimental

mixtures were stored at 40°C and 80°C during the night in an incubator without stirring. We found a milky precipitate during the extraction of the 1 M NaOH. Consequently, the concentration of the extract was measured by pre-filtration of the precipitate and by addition of the precipitate to the solution. This method was used only in the case of extraction at 40°C. After completing the extraction, the compounds were filtered by using a paper filter and the solutions obtained in such a way were measured by means of the ICP-MS method after the appropriate sample preparation methods. As can be seen from the results, solutions having a cobalt-concentration higher than that allowed by law were obtained after extraction in the case of 1 M NaOH, and, therefore, an experiment was performed on the filtered bottle flakes where a 48-hour extraction was performed again at 40°C by using a fresh dissolving agent. The *Lakanen-Erviö (LE)* extracting agent extracts ion-bound metals from the soil for which mobilization by the roots can well be modelled and the proportion that can potentially be taken by the plants through their roots can be subtracted.

Table 1. The solvents and extraction parameters

Solvents used	PET flakes contents and the extraction parameters
1 M NaOH	Temperature of solvents: T = 40°C and T = 80°C
1 M HCl	
5% ammonia solution	Extraction time: $t_{\text{extr}} = 48 \text{ h}$
Distilled water	
20% household vinegar	Mixing time: $t_{\text{mix}} = 16\text{-}17 \text{ h}$
Orange Juice (100%)	
Fruit brandy *	Incubation time: $t_{\text{incub}} = 31\text{-}32 \text{ h}$
Ethanol % (V/V): 49.986	
Density: 0.9302 g/cm ³	Mixing speed: $n = 500 \text{ rpm}$
1% solution of washing powder (Aro Univerzális Mosópor)	
10X dilution of liquid detergent (Diversey Tip Professional -Kézkímélő mosogatószer)	Amount of flakes used: $m = 10 \text{ g}$
100X dilution of liquid detergent	
	Amount of solvents used: $m = 100 \text{ ml}$
	*Density and ethanol content of fruit brandy were determined by Bálint Analitics Ltd.

The extract made by such an extracting agent shows the actual toxicity. This method can well be used to characterize the metal-contamination of contaminated soil- and waste-samples. The extracting agent we used is made as follows: The LE extracting agent contains 0.5mol/dm³ ammonium-acetate, 0.5 mol/dm³ acetic acid, and 0.02 mol/dm³ EDTA (ethylenediamine-tetra-acetic acid). Its pH is 4.65+/-0.05. 5.85 g of EDTA, 35 ml of 25% (m/m) ammonium-hydroxide and about 400 ml of water were measured in a flask with a volume of 1000ml. After the EDTA had completely dissolved in the ultrasonic tank, 57 ml of acetic acid was added to the compound.

Then it was diluted to approximately 950 ml with water. The pH-value of the solution could be set to a value of 4.5+/-0.05 by using 3 M ammonium-hydroxide or acetic acid as needed. For making the extract, a 5 g sample was added to 50 ml of extracting agent and then it was mixed for an hour by a shaking machine. *Dissolution-kinetic tests* were performed in order to know the kinetics of dissolution. In the course of planning the experiments, we decided to use two types of dissolving agents: one, fruit brandy, through which the largest quantity of cobalt could be dissolved without causing a chemical reaction and another, 1 M NaOH, by which a chemical reaction takes place while dissolving the largest quantity of cobalt. The extraction parameters of dissolution kinetics are identical to the parameters of the 48-hour extraction experiments (10 g powder, 100 ml of dissolving agent, $n=500\text{rpm}$) though the experiment was performed only at 40°C. In the course of the test, dissolution was started simultaneously in 6 compounds. Then the concentration of cobalt was investigated in the individual solutions at given time periods (8, 24, 32, 48, 72 and 104 hours). The testing method was the same for both the fruit brandy and the 1 M NaOH dissolving agents.

5. ICP-MS sample preparation for determining the cobalt-content of PET flakes

Following homogenization, a sample containing approximately 0.5 g cobalt was measured in a glass tube by using a tare scale and then ~5 ml of 68% nitric acid and ~4 ml of 30% hydrogen peroxide were added to it. This mixture was then digested by a microwave process using a Milestone Ultrawave ECR type microwave destruction autoclave programmed for $t=25$ min; the starting pressure was $p=40$ bar and the value of $T_{\max}=220^{\circ}\text{C}$. After the whole quantity of flakes had been digested (dissolved, got into the solution), the compound was diluted to 25 ml in a volumetric flask by using distilled water. The solution prepared in such a way was injected into the instrument. Owing to the organic-matter content of orange juice-, washing- and dishwashing solutions, a microwave digestion process was also applied. The method of preparing the individual samples was identical to the process used for preparing the PET flakes, though it was enough to use a lower quantity of destruction agents including the quantity of nitric acid and hydrogen peroxide as well. 5 g of the each of the extracts remaining after the other dissolving agents was measured in separate beakers by means of a tare scale and they were evaporated until dry. If no precipitation was experienced, the beakers were rinsed with 3 ml of 68% nitric acid and they were washed into volumetric flasks of a volume of 25 ml. If there was precipitation, the compound was distilled by adding 5 ml of 68% nitric acid and 3 ml of 30% hydrogen peroxide until 1-2 ml liquid remained on the bottom of the beaker. Then the compound was washed into a volumetric flask having a volume of 25 ml without residue by means of distilled water. Such an extract was, for example, the vinegar extract – in this case a brown precipitation was experienced. A blind sample and a background sample were both prepared during the preparation of the extracts. The blind sample was the clean dissolving agent (e.g. 1 M NaOH, fruit brandy, orange juice) used during the extraction process. It was necessary for investigating the quantity of cobalt in the given clean dissolving agent (c_{blind}). The background sample was the compound of materials used during the preparation which can “incidentally” be contaminated ($c_{\text{background}}$). The background sample consisted of 5 ml of 68% (m/m) nitric acid and 4 ml of 30% hydrogen peroxide diluted to 25 ml. So the result measured in the given extract (c_{extr}) had to be corrected in each case as follows: $c_{\text{korr}} = c_{\text{extr}} - c_{\text{blind}} - c_{\text{background}}$. Both the earlier and the further results are the concentration values corrected in accordance with the above formula (c_{korr}). During the preparation of the individual samples, an internal standard solution had been added in the given quantity before the volumetric flasks were filled to the given marking in such a way that after filling them to the given marking, the scandium, rhodium, and lutetium concentrations of solution was 1 ppb. All cases Agilent 7500 ICP-MS type mass-spectrometry instrument was used for measurement purposes.

6. Results of the extraction investigations

The results of cobalt dissolution tests performed by using different dissolving agents are shown in Table 2. The extraction experiments were performed at both 40°C and 80°C in order to reveal the effect of temperature increase regarding dissolution. In the course of the experiments performed at 40°C , a milk-white precipitation was experienced during digestion by nitric acid when preparing the extract obtained during the dissolution test performed by using 1 M NaOH. It was supposed that the precipitate could include some cobalt. So the cobalt-content of the extract was investigated in such a way that the precipitate was filtered and then the clean extract was prepared for an ICP-MS test. The other version of precipitate investigation was that the precipitate was digested by microwave destruction and later its solution was tested again.

6.1. Results of the 48-hour extraction experiments

Owing to the high degree of dissolution of 1 M NaOH, the dissolution test was performed again at 40°C in such a way that the extract was filtered after completing the 48-hour extraction (1/2) and a 48-hour extraction was performed again by adding fresh extracting agent to the filter cake that remained plastic flakes (2/2). The results of these experiments are also shown in Table 2. It was found that in the case of NaOH, in contrast to the other dissolving agents, not just surface dissolution takes place. Because PET is hydrolysed by NaOH, the dissolving agent can better diffuse into the structure of

material, so it can take in a higher quantity of cobalt into the solution than that experienced in the case of the other dissolving agents. The results of the dissolution tests are shown graphically in Fig. 1. The efficiency of extraction is determined by the percent of cobalt getting into the solution from the quantity of cobalt contained in the flakes used for extraction. It is calculated by the following relationship:

$$\eta_{\text{ext}} = ((0.1 \cdot c_{\text{corr}}) / (c_{\text{PET}} \cdot m_{\text{flakes}} / 1000)) \cdot 100 \quad (1)$$

Where, η_{ext} : efficiency of extraction as a percentage, c_{corr} : measured and corrected concentration of the given extract, c_{PET} : Cobalt-concentration of PET flakes, m_{flakes} : Mass of flakes used for extraction (~10 g).

Table 2. Results of the 48-hour extraction experiments

Solvent	Cobalt Concentr. 40°C (ppb)	Powder used for extraction 40°C (g)	Efficiency of extraction 40°C (%)	Cobalt Concentr. 80°C (ppb)	Powder used for extraction 80°C (g)	Efficiency of extraction 80°C (%)
1 M HCl	6.27	10.043	0.17	9.04	10.018	0.25
1 M NaOH	92.92	10.034	2.52	-	-	-
1 M NaOH precipitate	112.15	10.034	3.04	170.77	10.017	4.63
5% aqua ammonia	11.17	10.036	0.30	11.20	10.024	0.30
Distilled water	0.97	10.038	0.03	1.57	10.032	0.04
20% acetic acid	9.05	10.024	0.25	7.67	10.022	0.21
Orange juice	8.66	10.033	0.23	5.88	10.037	0.16
Fruit brandy	11.58	10.004	0.31	17.25	10.031	0.47
Washing powder 1%	4.35	10.011	0.12	7.31	10.001	0.20
Detergent 10X	3.25	10.007	0.09	9.93	10.027	0.27
Detergent 100X	2.68	10.021	0.07	7.27	10.018	0.20
1 M NaOH 1/2	118.37	10.030	3.21	-	-	-
1 M NaOH 2/2	183.97	-	-	-	-	-

It can be concluded from the results of the experiments seen in Fig. 1. that the dissolved quantity was not dangerous for consumers as far as the dissolving agents used in the household (water, fruit brandy, and orange juice) are concerned. The quantities of the agents dissolving under the influence of the higher temperature and shaking during delivery do not have any dangerous effect either. Certain unwholesome effects can only be observed in case of an intake of a quantity of 13.3-58.9mgCo/ kgbw, and—even in the worst case—the dissolution of merely 17.25 ppb of orange juice could be experienced. Therefore, it is considered that the quantity of intake/day cannot be more than 10-20 µg in case of consumption of an even a higher quantity; here it is necessary to take into consideration the fact that a surface dissolution takes place in the bottles while the flakes used for dissolution during the experiments have got a higher specific surface. The RDA for cobalt/day is 0.1 µg for an adult of average body weight (in the form of 2.4 µg B12), which is obtained for the human organism from vitamin B12 being the only source of cobalt.

6.2. Results of the Lakanen-Erviö dissolution test

The cobalt-concentration was 0 ppb in all the three Lakanen-Erviö extracts made simultaneously. As the LE extracting agent does not extract cobalt from PET, it can be stated that no serious cobalt contamination can be caused in the soil by the PET plastic flakes containing cobalt. Cobalt releases from the plastic matrix during the bio-degradation of PET, so it can continuously dissolve into the soil

and into the ground water over the centuries. In addition, it can be supposed that the bottles being in landfills biodegrade faster owing to the more intensive circumstances: the pH of leachate of older landfills is characteristically alkaline, and, moreover, a higher temperature (40-70°C) can prevail in them during the bio-degradation of more waste-mass.

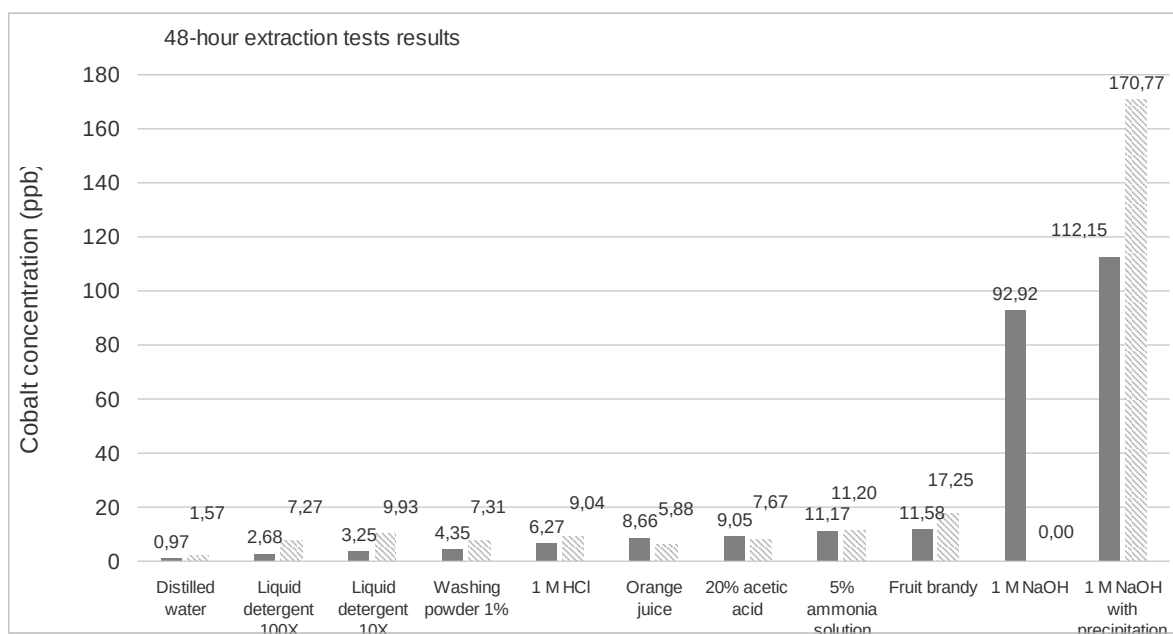


Figure 1. Cobalt extraction results in different domestic solvents (investigation performed at 40°C- solid, at 80°C – stripped)

6.3. Results of the dissolution-kinetic tests

As can be observed in Fig. 1, the dissolution of merely 0,97-11,58 ppb of dissolving agent could be experienced even in the case of fruit brandy considered as a dissolving agent that dissolves the highest quantity of cobalt without a chemical reaction in 40°C. The chemical reaction speed itself can well be observed in the case of the NaOH dissolving agent in Fig. 2.

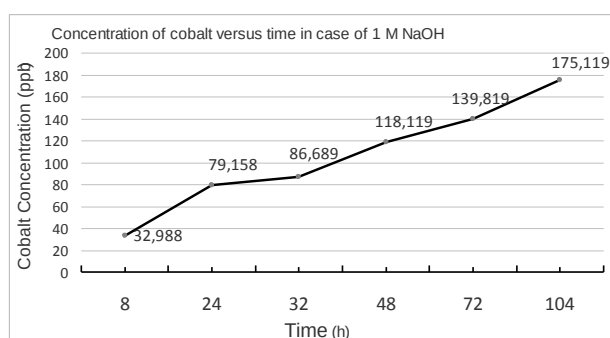


Figure 2. Dissolution-kinetic in the case of 1 M NaOH dissolving agent

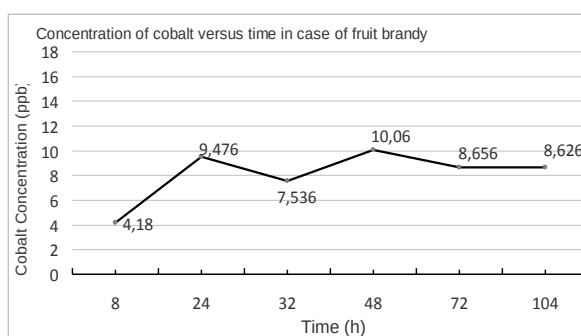


Figure 3. Dissolution-kinetic in the case of fruit brandy

PET is hydrolysed by soda-lye, so the surface structure of the individual grains is decomposed by it. In this case the dissolving agent is able to penetrate into the internal structure of the grains, and the dissolution speed keeps a nearly constant value over time as can well be observed in Fig. 2. The surface dissolution can also well be seen in Fig. 3 in the case of fruit brandy. Cobalt-segregation stops increasing in the solution after 24 hours. The dissolving agent is not able to penetrate into the internal

structure of plastic grains after reaching a maximum concentration of 10,06 ppb, so further dissolution stops. Minor variations can be experienced between the individual measurement points in Fig. 3.

7. Summary

In our paper, cobalt-dissolution tests of PET bottles having an active oxygen scavenger layer were performed by dissolving agents used in the household. Similar to the first step of recycling of empty PET bottles, a sample was ground and swelled in order to estimate the extent of cobalt-dissolution from the additive material containing cobalt. In the course of the experiments using ICP-MS investigation technology, it was found that the average cobalt-content of three-layered PET-flakes containing Amosorb active oxygen scavenger was 36.805 mg/kg. The fruit brandy and the orange juice contained in the bottles were the two most successful agents dissolving cobalt without chemical reactions. The concentration of cobalt-extracts was 11.58 ppb and 8,66 ppb respectively with these agents. 1 M NaOH was the agent dissolving the highest quantity of cobalt. As a consequence of the hydrolysis of PET, a continuous dissolution over time could be experienced here when this soda agent penetrates into the grains of flakes. With the exception of NaOH, the dissolution of cobalt did not increase considerably by changing the temperature. It can be concluded from the aforementioned facts that the quantity of dissolved cobalt was not dangerous for consumers as far as the dissolving agents used in the household are concerned and applied by us in the form of flakes. Namely, some unwholesome effects could only be observed in the course of certain animal experiments when a quantity of 13.3-58.9 mgCo/kgbw (equal approximately 13.3-58.9 ppm of Cobalt) was taken. More dangerous effect could be arising in the case of natural water containing multi-layered PET flakes or bottles under long term deposition. We suggest that an individual collection or special recycling process technology need to be performed on these PET bottles. If not, the environmental impact increases under the influence of such active oxygen scavenger layered PET bottles. However, it can be stated also that the material of these kinds of PET bottles decomposes faster and the cobalt releases if they are placed in a communal landfill owing to the higher temperature and the presence of an alkaline solvents.

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