JEE Journal of Ecological Engineering

Journal of Ecological Engineering 2023, 24(3), 169–177
https://doi.org/10.12911/22998993/158381
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Received: 2022.12.04
Accepted: 2023.01.05
Published: 2023.01.15

Analysis of the Possibility of Heavy Metal Ions Removal from Aqueous Solutions on Fruit Pomace

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ABSTRACT

Pomace from apples, grapes, blackcurrants, and oranges is a waste product of fruit processing, which is formed during the production of juices. Pomace is a rich source of biologically active compounds such as polyphenols, carotenoids, or vitamins. They also contain pectin, cellulose, lignin, proteins, and minerals. All these components, apart from having many beneficial properties for human health, also show sorption properties towards heavy metal ions. Therefore, this study aimed to evaluate the possibility of removing lead (II) and cadmium (II) ions from aqueous solutions by adding apple, grape, black currant, and orange pomace as well as to determine the adsorption efficiency of selected heavy metals. The studies were carried out in model systems. The results of the research showed that lead (II) and cadmium (II) ions are adsorbed on fruit pomace. The highest removal of cadmium (II) ions was observed in orange pomace and of lead (II) ions in apple pomace. The maximum tested cadmium ions absorption capacity was: 0.117235 mg/g in the case of freeze-dried apple pomace; 0.08618 mg/g in the case of freeze-dried currant pomace; 0.21915 m/g in the case of freeze-dried grape pomace and 0.29549 mg/g in the case of freeze-dried orange pomace. On the other hand, the maximum absorption of lead ions was: 0.457 mg/g in the case of freeze-dried apple pomace; 0.442 mg/g in the case of freeze-dried currant pomace; 0.3445 mg/g in the case of freeze-dried grape pomace and 0.421 mg/g in the case of freeze-dried orange pomace. On the basis of the performed measurements, it can be concluded that the tested waste can potentially be used as a cheap biosorbent for removing heavy metal ions from dilute aqueous solutions.

Keywords: fruit pomace, adsorption, heavy metals.

INTRODUCTION

In recent years, there has been a progressive degradation of the environment. Insufficiently treated wastewater, dust, waste gases, and municipal and industrial waste have caused an increase in the content of heavy metals in surface and groundwater over the past decades. This is an unfavorable and dangerous phenomenon, because compounds of such elements as copper, chromium, cadmium, or lead are not biodegradable and accumulate in living organisms, thus passing into the trophic chain and posing a threat to human health and even life. Moreover, their presence in rivers, lakes or water reservoirs disturbs the biological balance, slows down the self-purification processes as well as hinders water purification and treatment at water treatment stations (Krol and Nawirska 2003; Dixt et al. 2015). Currently, the most commonly used methods for removing heavy metals from aqueous solutions are chemical and electrochemical precipitation or ion exchange processes.

Because more and more emphasis is placed on the conscious management of environmental resources and sustainable development in all activities, new solutions and technologies are sought to remove heavy metals, including lead and cadmium from water. Moreover, new solutions and technologies are sought to remove heavy metals, including lead and cadmium, from water. One such method is the use of the biosorption process, which consists of adsorption, surface precipitation, ion exchange, and complex formation. This method can be an alternative to using expensive and conventional methods to remove metals from drinking water (Ince and Ince 2017). The most popular adsorbents are zeolites, silicas, activated carbon, and chitosan. However, new materials of most favorably plant origin are still being sought as economical, ecological, and easily accessible sorbents for the removal of pollutants from aqueous solutions (Tomczak and Szczerkowska 2010). Waste from agri-food processing, including the fruit and vegetable industry, can be used to carry out the biosorption process. The management of this waste is part of the issues of environmentally friendly technologies and processes, the circular economy, as well as the use of effective and economical (low-cost) materials (Volesky 2001). Because fruit pomace contains cellulose, hemicellulose, and lignins, it can be characterized by good sorption properties. In addition, fruit pomace constitutes about 25% of the waste mass from fruit and vegetable processing; thus, it may be a readily available and cheap source of natural sorbents.

There are several examples in the literature of fruit and vegetable processing waste being used as adsorbents for heavy metal removal (Sheibani et al. 2012; Sulyman et al. 2017; Kalak et al. 2020). However, there are few reports of the use of pomace in the production of juices. An example of application is the results obtained by a research group (Hegazy et al. 2021), which conducted studies on the adsorption of iron ions Fe^{2+} and manganese ions Mn^{2+} on olive pomace and seed husks of a plant called moringa. They showed that the percentage removal of Fe^{2+} and Mn^{2+}, by 5 grams of olive pomace was 83% and 91%, at pH 5 and ideal time 120 min. On the other hand, the percentage removal of Fe^{2+} and Mn^{2+} ions was 80.5% and 93%, at 5 grams for moringa seed husks. Studies by Uzunkavak et al. (2019) proved that it is possible to remove Cd^{2+} and Pb^{2+} ions on olive pomace. The maximum adsorption capacity was obtained at the level of about 64 mg/g for lead ions and 20.5 mg/g for cadmium ions. On the other hand, Aziz and his research group demonstrated that moringa seeds, banana peel, and their combination could be used as natural adsorbents in water treatment, especially for the removal of Pb^{2+} , Ni^{2+} and Cd^{2+} ions from drinking water (Aziz et al. 2016). The results showed that the combined biomasses were able to meet the WHO standards for concentrations of Pb, Ni, and Cd.

Therefore, this study aimed to investigate selected fruit pomace from juice production as potential sorbents as well as evaluate the possibility of removing lead and cadmium ions from aqueous solution sand to determine the adsorption efficiency of selected heavy metals. The adsorption properties depend on pH and temperature, but the article investigates the properties of different adsorbers under the same conditions. Attention was focused on the evaluation of individual fruit pomace, which differs in the type of fruit and the method of drying.

MATERIALS AND METHODS

The fruit pomace came from the Podlaskie Voivodeship, the fruit and vegetable processing plants from the production of fruit juices. The pomace used in the study was an apple, blackcurrant, white grape, and orange, which were subjected a to drying process, including convection

Figure 1. Fruit pomace from left to right: oranges, blackcurrants, grapes, and apples

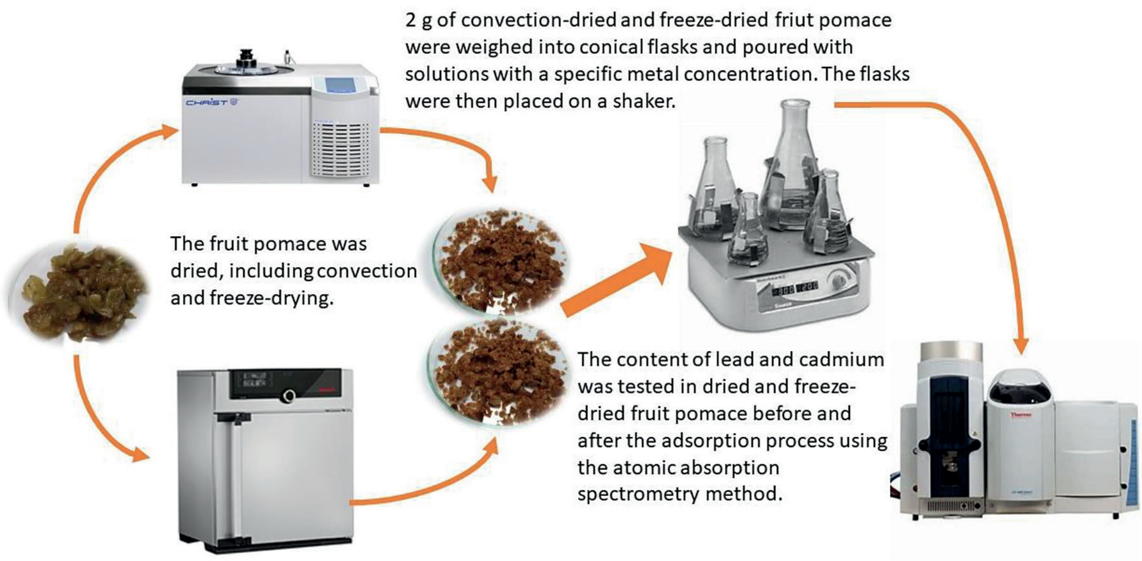

Figure 2. The scheme of performing the experiment

and sublimation drying. Figure 1 shows the fruit pomace used in the experiment.

To investigate the sorption of heavy metal ions on fruit pomace, an experiment was performed and aqueous solutions of a specified concentration of 10 mg/dm^3 for lead and 4 mg/dm^3 for cadmium were prepared in 50 ml volumetric flasks. Then, 2 grams of convection-dried and freeze-dried pomace from apples, blackcurrants, white grapes, and oranges were weighed into conical flasks for each element, respectively, in triplicate. The test pomace was flooded with solutions of the specified metal concentration. The flasks were then placed in a shaker and the solutions were sampled after 15, 30, 45, 60, and 75 minutes. The determination was carried out up to 75 min, because according to (Krol and Nawirska 2003), the pomace loses its sorption properties after about 60 min. Furthermore, the content of lead and cadmium was examined in dried and freeze-dried fruit pomace before and after the adsorption process. Figure 2 shows the consecutive stages of the experiment.

The heavy metal content examined was determined by atomic absorption with flame atomization using a Thermo Scientific iCE3500 atomic absorption spectrometer with deuterium background correction. For the determination of each of the elements, their standard solutions were prepared in flasks with a capacity of 50 cm^3 enabling the plot of a standard curve consisting of 3 measuring points each. The reference sample was distilled water (Szatyłowicz and Skoczko 2019). Table 1 shows the limit of detection and limit of quantification of heavy metals, characterized by an iCE 3500 spectrometer (Thermo Scientific's AAS User Manual 2013). In parallel, the matrix reference material TMDA 54.6 (lot 1219) by company Labmix24 Germany was analyzed to verify the correctness of the method (Table 2) Trace element matrix Reference Materials (RMs) are made with filtered and diluted Lake Ontario water and are preserved with 0.2% nitric acid.

Table 1. The instrumental detection limits (IDL) and instrumental quantification limits (IQL) for the iCE 3500 spectrometer [mg/dm^3] (Thermo Scientific's AAS User Manual 2013; Szatyłowicz and Skoczko 2019)

Element	Model [mg/dm^3]	IQL [mg/dm^3]
Pb	0.0130	0.070
Cd	0.0028	0.013

Table 2. Comparison of measured and certified concentrations in TMDA 54.6 [mg/dm^3]

Element	TMDA 54.6		AAS		Dev*
	Concentration [mg/dm^3]	±SD [mg/dm^3]	Concentration [mg/dm^3]	±SD [mg/dm^3]	[%]
Pb	0.490	0.029	0.523	0.054	+ 6.31
Cd	0.157	0.008	0.163	0.010	+ 3.68

* **Note:** Relative difference between measured and certified concentration: 100%·(cm–cc)/cc

This fortified bulk RM has concentrations in the high range (Szatyłowicz and Skoczko 2019).

Equation 1 was used to calculate the removal efficiency of heavy metal ions (R) from model solutions (Krol and Nawirska 2003; Skoczko and Szatyłowicz 2018):

$$R = \frac{C_o - C_t}{C_o} 100\% \quad (1)$$

where: C_o – initial concentration [mg/dm³];
C_t – concentration after time [mg/dm³].

Furthermore, the metal binding capacity (Ao) of the pomace was calculated from equation 2 (Krol and Nawirska 2003; Skoczko and Szatyłowicz 2018):

$$A_o = V\frac{(C_o - C_k)}{m} \quad (2)$$

where: A_o – the capacity of the pomace to bind ions [mg/g];
C_k – equilibrium concentration of the metal [mg/dm³];
C_o – initial metal concentration [mg/dm³];
V – the volume of the solution [dm³];
m – pomace mass [g].

RESULTS AND DISCUSSION

The conducted observations demonstrate that the tested fruit pomace was characterized by different effectiveness in removing lead and cadmium from aqueous solutions (Figures 3, 4). It was observed that freeze-dried pomace had higher efficiency of heavy metal removal from model solutions. It may be related to the greater porosity of these types of materials used as sorbents (Tomaczak and Szczerkowska 2010). Moreover, according to Shishehgarh et al. (2002) and Chakraborty et al. (2006) the porous microstructure of freeze-dried materials is characterized by a very high water absorption capacity, but low resistance to mechanical damage. Convection drying, on the other hand, causes very high shrinkage and reduction of porosity of the material as well as deterioration of the sorption effect (Ciurzyńska et al. 2013).

The orange pomace was the most effective in removing cadmium ions among the freeze-dried pomace, the effectiveness of which increased in direct proportion to the time of contact with the aqueous solution. This relationship was linear and the coefficient of determination R2 was equal to 0.94. For this pomace, more than 70% efficiency of

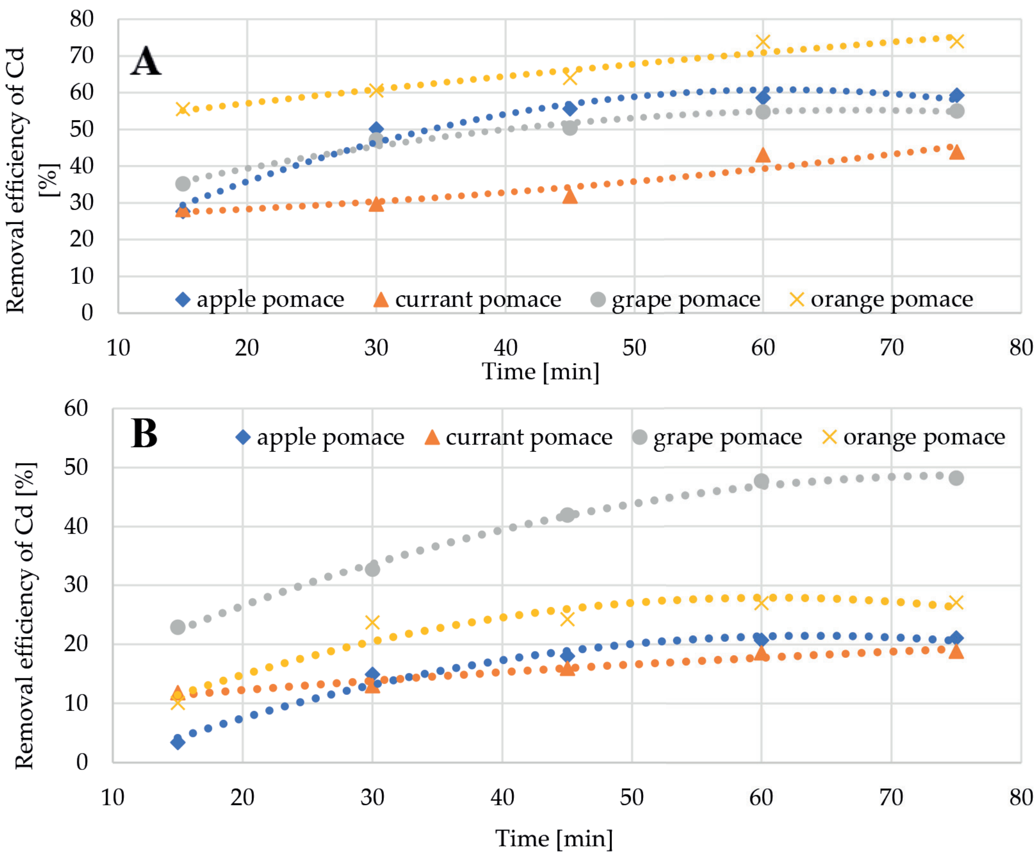

Figure 3. The efficiency of cadmium ion removal on fruit pomace: A – freeze-dried, B – conventionally dried

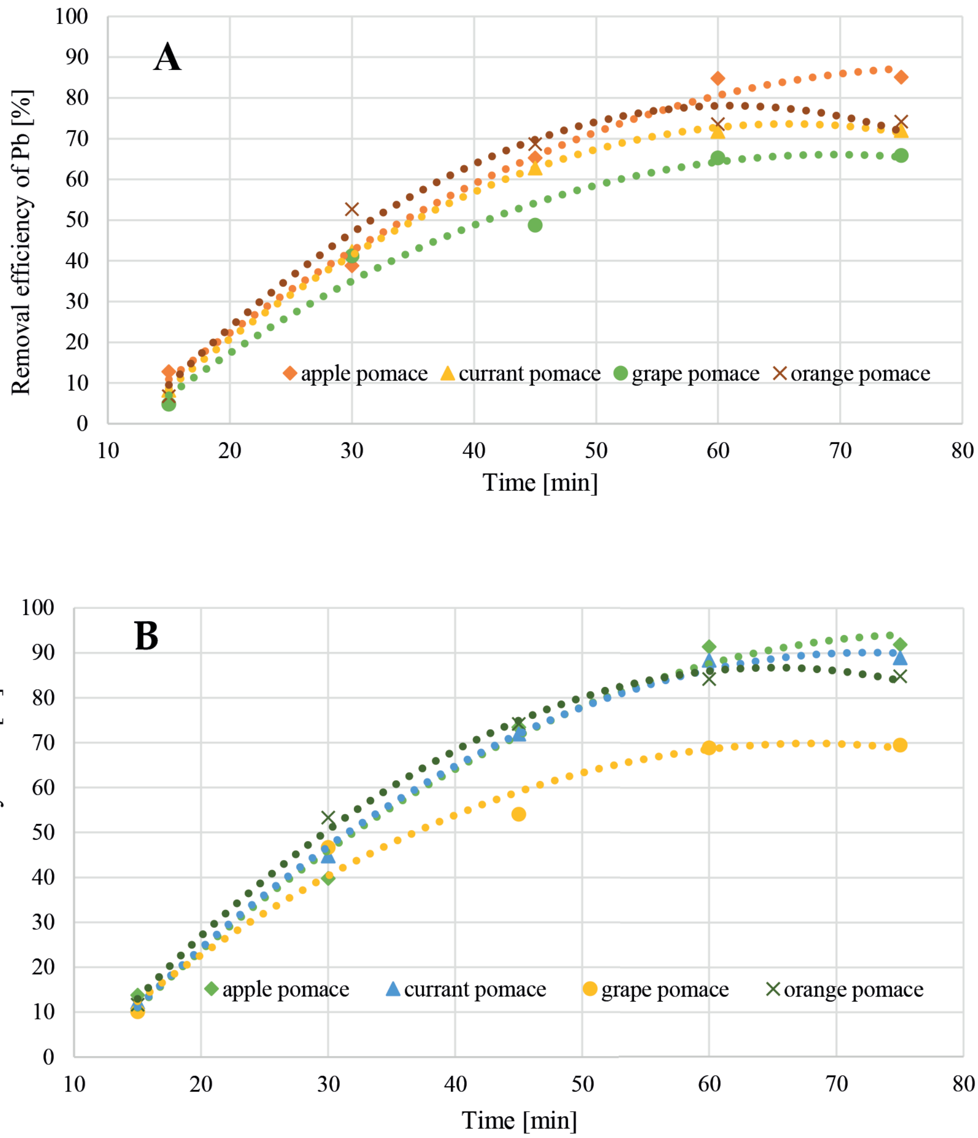

Figure 4. The efficiency of lead ions removal on fruit pomace: A – freeze-dried, B – conventionally dried

cadmium removal from the aqueous solutions tested was observed, with the highest efficiency of this process, since more than 50%, already observed after 15 minutes. On the other hand, blackcurrant was characterized by the lowest efficiency among the sublimation dried pomace, which after 70 minutes achieved only 40% cadmium removal efficiency. Moreover, non-freeze-dried currant pomace represented the lowest effectiveness. In the case of convection-dried pomace, the grape had the highest adsorption efficiency. Most of the tested freeze-dried fruit pomace showed more than a 50% better efficiency in removing cadmium (II) ions from the solutions than the convection dried pomace. The exception was the grape marc, the adsorption activity of which remained at a similar level.

Comparing the cadmium adsorption efficiency (Figure 3) on the tested fruit pomace with the lead adsorption efficiency (Figure 4), a much higher adsorption affinity for the lead was observed. Similar results were obtained by (Krol and Nawirska 2003) where the best-bound metal was lead, the removal effect of which exceeded 98%. Farinellaet et al. (2008) also displayed a better affinity of grape pomace for lead ions than cadmium. Lead adsorption in the conducted research was slower but more effective. This phenomenon is related to the value of the Stokes rays for both metals. The radius of hydrous lead is 0.297 nm, while for cadmium it is equal to 0.393 nm. A smaller Stokes radius favors a better adsorption capacity of a given metal on the surface (Ring 1996). The Sciban research group (2007),

Table 3. The efficiency of heavy metal ion removal from aqueous solution after 75 minutes on selected fruit pomace

Pomace	Type of drying	Cd	Pb
		[%]	
Orange	sublimation	74	85
	convection	27	74
Grape	sublimation	55	70
	convection	48	66
Apple	sublimation	59	92
	convection	21	85
Currant	sublimation	44	89
	convection	19	72

which tested the adsorption of cadmium, copper, and lead ions on sawdust, found that the adsorption of cadmium ions was less effective than the adsorption of copper and zinc. Sprynskyy et al. (2006), who investigated the adsorption of Pb^{2+}, Cu^{2+}, Ni^{2+}, and Cd^{2+} on clinoptilolite, proved that among the examined metals from one-component solutions, lead adsorption was much more effective than cadmium adsorption. The mentioned examples confirm the influence of the atomic radius on the adsorption power by comparing different sorbents.

The lead ions were adsorbed most efficiently on freeze-dried apple pomace. In the case of lead, the dependence of the amount of the removed ion on the pre-treatment of pomace was also observed. Grape pomace was characterized by the lowest efficiency of removal of this metal, which after 75 minutes was estimated at 70% (Table 3).

The capability of removing heavy metal ions by fruit pomace increased with prolonged contact time (Table 3). Comparing the differences between the removal efficiencies of cadmium and lead ions from aqueous solutions, it was found that the efficiency curves for cadmium ion removal on both freeze-dried and convection-dried pomace had a more rectilinear course. The percentage of cadmium removal on freeze-dried pomace increased rapidly during the first 15 min and slowly increased over one hour. Equilibrium biosorption was established after about 60 min and the maximum removal efficiency was over 40% for freeze-dried currants and over 70% for freeze-dried orange peels. However, no sudden increase in removal efficiency was observed with a further extension of contact time (> 15 min). Further observations showed that the maximum sorption capacity of cadmium by fruit pomace was 29.5 mg/100g for freeze-dried orange peels. (Figure 5).

At the same time, it should be noted that in the case of non-lyophilized pomace, the initial cadmium removal efficiency was much lower, while the equilibrium state was also observed

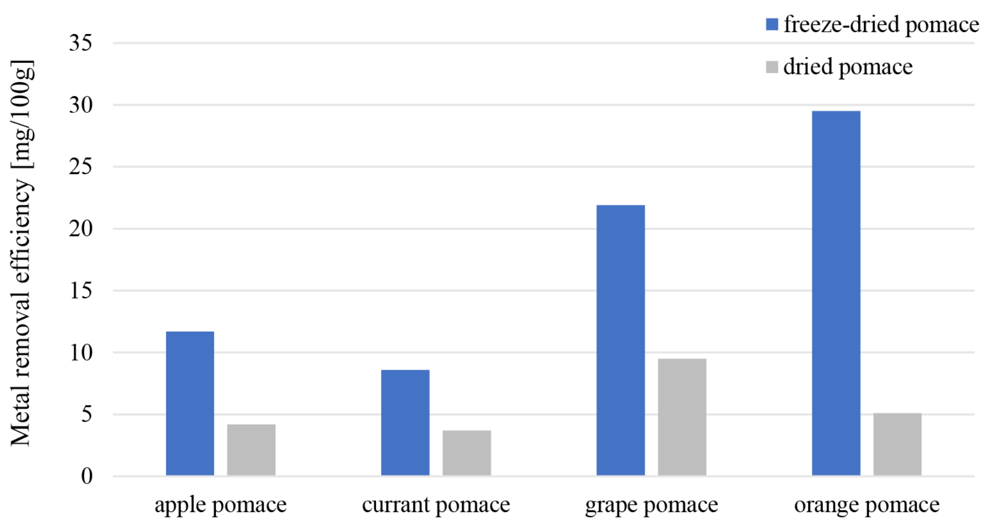

Figure 5. The cadmium binding capacity of the tested pomace

after about 60 min. Moreover, the cadmium-binding capacity of dried pomace was different, in this case, the grape pomace was characterized by the highest capacity (9.5 mg/100g; Figure 5). The analysis of the efficiency of lead removal from aqueous solutions is different. The course of the curve describing the efficiency of removal of this metal was not linear and it was observed that after 15 minutes the efficiency of this process was significantly lower than in the case of cadmium, and after another minutes, it enhanced significantly. The equilibrium of biosorption also in this case can be determined at about 60 minutes. Similar observations were presented in the work of Iqbal et al. (2009), where the sorption kinetics for both metals (cadmium and lead) on mango peels was rapid, reaching equilibrium at 60 min.

The curves of this process are similar for freeze-dried and convection-dried fruit pomace. At the same time, a significantly higher ability of pomace to bind lead than cadmium was observed, and the differences in this respect between pomace subjected to different pretreatments were much smaller (Figure 6).

On the basis of the above results, it can be assumed that there are two stages of biosorption of cadmium and lead ions. The first stage is a short period of 15 to 30 minutes and the second stage is until equilibrium is established. As reported by Chen et al. (2021) the removal of copper ions from solutions by the yeast species Pichia pastoris also occurs in two stages, as this may be related to the fact that the cell wall of the adsorbent is formed by different macromolecules and these complex structures provide potential binding sites. Therefore, the structure of sorbents influences the efficiency and kinetics of the heavy metal adsorption process. Considering fruit pomace, it is the proportion of basic components such as pectin, cellulose, and lignin that determines the course of the sorption process, as they show strong sorption properties. The research carried out by Nawirska and Oszmiański (2001) allowed arranging a series in which particular fractions bind metals. In the case of cadmium ions and apple pomace, these are pectin>lignin>hemicellulose>cellulose. In the case of lead binding, the series of binding fractions are as follows: polyphenols>pectins>hemicellulose>cellulose>lignins.

The results obtained confirm the observations made by other authors about the possibility of using fruit pomace to remove metal ions from contaminated water or wastewater and these observations are part of the research conducted in recent years. Pavan et al. (2008) studied the ability to remove lead ions from aqueous solutions using biosorbents. They found that citrus peels have a sorption capacity of 112 mg/g. Moreover, Farinella et al. (2008) investigated the possible use of grape pomace to adsorb Cd^{2+} and Pb^{2+} ions from wastewater. They used optimized pH and a contact time of 5 minutes in their research. On the basis of the obtained results, it was calculated that the adsorption capacity was 0.774 mmol/g for cadmium ions and 0.428 mmol/g for lead ions. In the studies of Iqbal et al. (2008) mango peel was assessed as a new sorbent for removing Cd and Pb ions from an aqueous solution, the maximum sorption capacity of which was estimated at 68.9 for cadmium ions and 99 mg/g for lead ions. On the other hand, Anwar et al. (2010) showed that remains of lemon and orange are better suited to

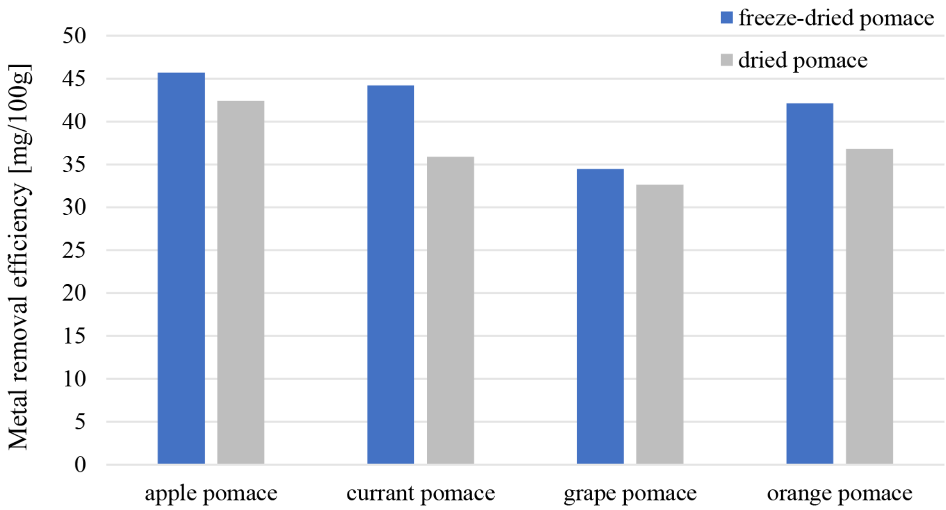

Figure 6. The lead binding capacity of the tested pomace

remove lead ions. On the other hand, banana peel was found to be better suited for the removal of cadmium ions. Moreover, the adsorption of Pb and Cd ions on banana peels was investigated and it was found that it is 5.71 for lead ions and 2.18 mg/ g for cadmium ions.

Summarizing the obtained results and review of the literature, it can be concluded that the global concerns about environmental pollution made it necessary to evaluate waste from fruit processing and vegetables as potential biosorbents. The results of numerous studies of these bio-waste prove that, in addition to being widely available, they have fast kinetics and significant adsorption capacity for heavy metals, for example from aqueous solutions containing high concentrations of heavy metals. However, to better understand the adsorption process of heavy metals on fruit pomace, additional research is needed on process parameters such as optimal temperature and environmental pH, and the structure of potential adsorbents. According to Patel (2012), further research could make pilot or industrial scale implications a sustainable reality. Furthermore, the proposed biosorbents could become an alternative method for removing contaminants from aqueous solutions.

CONCLUSIONS

On the basis of the research, it can be concluded that the effectiveness of the removal of lead and cadmium ions increases along with the contact time of the pomace with the contaminated aqueous solution. However, it should be remembered that taking into account the kinetics of the process, after reaching the state of equilibrium, heavy metal ions may be re-released into the treated solutions. The pre-treatment of the pomace as sorbents influences the efficiency of the heavy metal biosorption process. Among the examined pomace, freeze-dried pomace had the best sorption properties. Orange pomace was the most effective for cadmium adsorption, and apple pomace in the case of lead. Apple pomace, regardless of the drying method, showed the highest efficiency in removing lead ions, which was 85% for convection-dried pomace and 92% for freeze-dried pomace. Fruit pomace as a by-product of agri-food processing can be used in the processes of removing heavy metal ions from aqueous solutions. Furthermore, the use of fruit pomace for the removal of heavy metals from aqueous solutions can be an alternative method for its purification. This material can compete with other sorbents due to its ease of obtaining, low cost, good results, as well as economic and ecological effects.

According to the authors, one of the reasons for the increased adsorption of metal ions is the higher content of bioactive compounds in the press cake preserved by lyophilization compared to the preservation by conventional drying methods. Metals together with bioactive compounds form complexes, and chelates, which results in their increased sorption by lyophilized pomace, containing more bioactive compounds than conventionally dried pomace (Olesinska et al. 2018). In addition, freeze-dried fruit or pomace are characterized by a lower material density and a faster adsorption rate, which indicates high porosity and slight structural damage compared to the material fixed by thermal drying (Janiszewska et al. 2013).

Acknowledgements

The research was carried out as part of work no. WZ/WB-IIŚ/3/2020 and WZ/WB-IIŚ/2/2021 at the Bialystok University of Technology and financed by a subsidy provided by the Minister of Science and Higher Education

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