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Zinc adsorption from aqueous solution using lemon, orange, watermelon, melon, pineapple, and banana rinds

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ABSTRACT

In this study, six fruit rinds, chemical activated using $ZnCl_2$ and carbonized by microwave, were used as biosorbent to remove zinc from aqueous solution. Fruit kind, microwave treatment time and adsorption contact time effects on zinc biosorption yield were examined. The characterization of raw materials and synthesized biosorbents were realized by Fourier-transform infrared spectroscopy (FT-IR). The dried fruits rinds were activated for 2 or 4 h using $ZnCl_2$ and carbonized for 1, 2, 3 or 4 min at 800 W microwave power. Zinc biosorption yield was determined using ultraviolet-visible spectroscopy (UV-Vis). Maximum zinc biosorption capability was determined as 39.18 mg/g for lemon, 35.78 mg/g for orange, 14.76 mg/g for watermelon, 13.06 mg/g for melon, 12.2 mg/g for pineapple and 9 mg/g for banana rinds. The results indicate that the six fruits rinds can be used for zinc adsorption from aqueous solution as cost-effective and environmentally friendly biosorbent with high adsorption efficiency.

Key words: adsorption, biosorbent, fruit rind, microwave, zinc

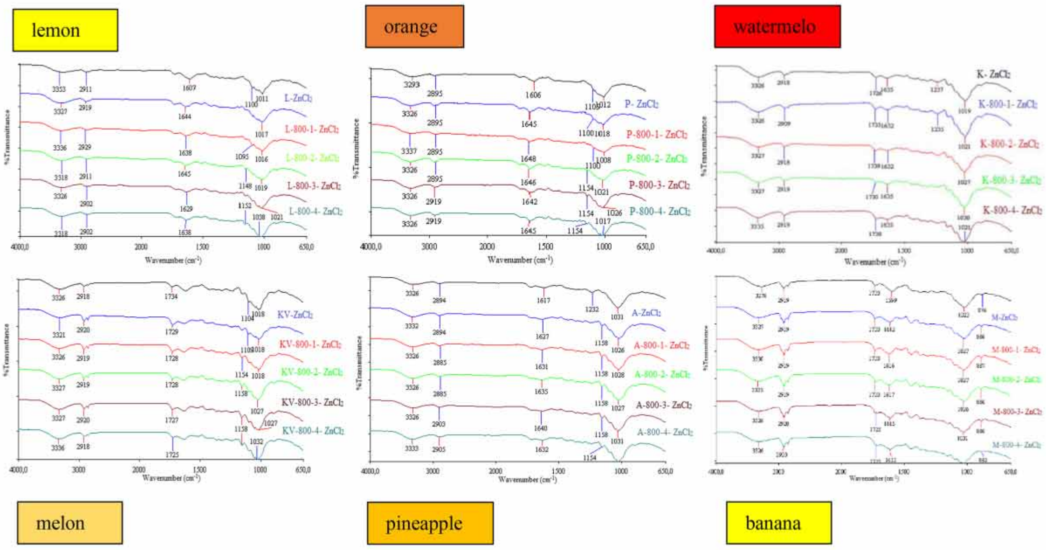
HIGHLIGHTS

- Fruit rinds were used as biosorbent to remove zinc from aqueous solution.
- Fruit rinds were chemical activated using $ZnCl_2$ and carbonized by microwave.
- Raw materials and synthesized biosorbents were characterized by FT-IR.
- Zinc biosorption yield was determined using UV-Vis.
- Cost-effective and environmentally friendly biosorbent with high adsorption efficiency was obtained.

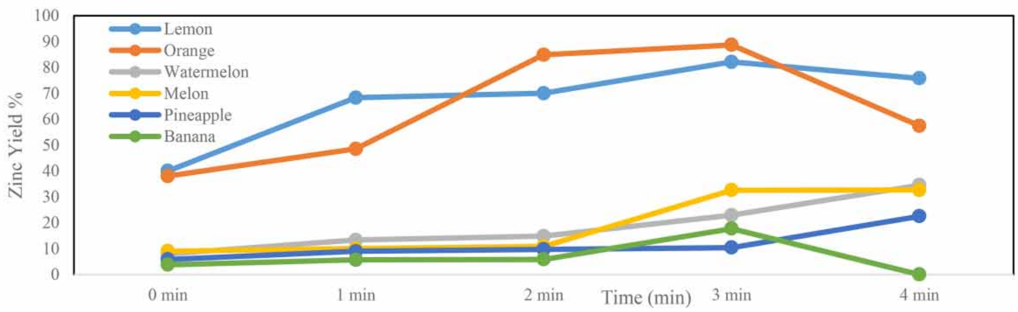
GRAPHICAL ABSTRACT

Weight loss of dried peels after microwave processing.

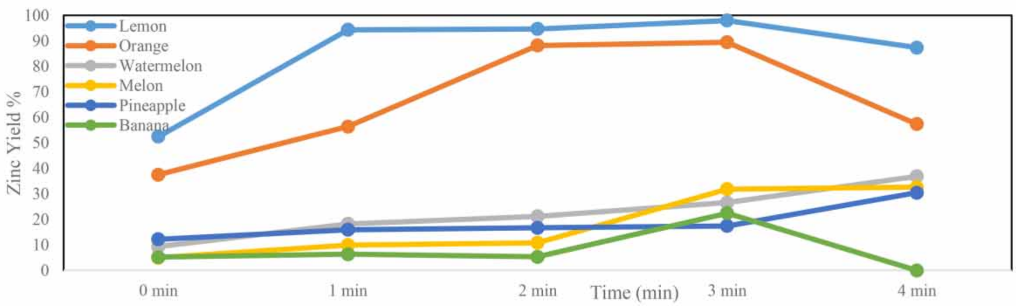
Adsorbent	Weight loss (%)			
	800 W-1 min	800 W-2 min	800 W-3 min	800 W-4 min
Lemon	3	9	17	28
Orange	4	10	22	28
Watermelon	0	6	11	27
Melon	3	7	14	30
Pineapple	2	4	9	16
Banana	4	9	12	26



FT-IR spectra of the dried rinds, and of the rinds activated with $ZnCl_2$ and microwaved for 1, 2, 3, 4 minutes



Microwave pretreatment time effects on Zinc adsorption yield (2h)



Microwave pretreatment time effects on Zinc adsorption yield (4h)

INTRODUCTION

Zinc has a significant role in the immune system at low concentrations. On the other hand, it causes a serious risk and threatens the environment and human health when it exceeds the permissible limits. Maximum allowable zinc limit by the World Health Organization in surface water, groundwater, and drinking water is 0.01, 0.05, and 3 mg/L, respectively (Bazana *et al.* 2019; Turkmen Koc *et al.* 2020; Dawn & Vishwakarma 2021; Jayakumar *et al.* 2021; Kumar & Pakshirajan 2021). Lots of methods have been studied for heavy metal removal from aqueous solution like adsorption, chemical precipitation, ion exchange, membrane filtration, electrocoagulation and electrodeposition (Altıntıg *et al.* 2021; Badmus *et al.* 2021; Saleh 2021). Biosorption is an attractive method due to large availability, low cost, operation facility and high heavy metal adsorption efficiency of the adsorbents. Different wastes are generated from the various sources such as domestic waste, agricultural waste, fertilizer waste, industrial waste, animal waste and marine waste. Agricultural waste rinds, being natural, economic and eco-friendly biosorbent, were utilized in the adsorption of water contaminant and to decrease the pollution grade. Pore size, specific surface area, surface functional groups and pH are the main characteristics that determine the biosorbent's performance (Bhatnagar *et al.* 2013; Bhatnagar *et al.* 2015; Wang *et al.* 2016; Romero-Cano *et al.* 2017; Meseldzija *et al.* 2019; Herrera-Barros *et al.* 2020; Dawn & Vishwakarma 2021). Most researchers have tried zinc adsorption from aqueous solution utilizing different agricultural-based biosorbents, like tangerine peels (Abdić *et al.* 2018); green marine macro algae, *Caulerpa scalpelliformis* (Jayakumar *et al.* 2021); Lagenaria siceraria peel (Dildar *et al.* 2018); Scots pine (*Pinus sylvestris* L.) and Silver birch (*Betula pendula*) (Komkiene & Baltrenaite 2016); orange, pineapple and pomegranate peels (Turkmen Koc *et al.* 2020); watermelon rind (Liu *et al.* 2012); sunflower, potato, canola and walnut shell residues (Feizi & Jalali 2015); almond shell (Çoruh *et al.* 2014); walnut shell (Segovia-Sandoval *et al.* 2018); tea waste (Wen *et al.* 2017); potato peel, lemon peel, orange peel, watermelon peel, tomato peel, coffee waste, apple peel, banana peel, decaf coffee waste, eggplant peel, carob peel and grape waste (Massimi *et al.* 2018); watermelon rind (Bhattacharjee *et al.* 2020); orange peel (Santos *et al.* 2015); and muskmelon peel (Khan *et al.* 2017). Watermelon is one of the most widely eaten fruit around the world. The edible part of the fruit composes nearly 70% of the total mass and the rind composes nearly 30% (Liu *et al.* 2012; Bhattacharjee *et al.* 2020). The peel of the matured melon is one of the common kitchen bio-wastes produced in large amounts each year (Huang & Zhu 2013; Khan *et al.* 2017; Karthikeyan *et al.* 2020). The rinds of citrus fruits like lemon and orange, which are generally wasted, are used by juice processing industries. Approximately 75% of orange fruit and 50% of lemon fruit become a byproduct or waste after juice extraction. Physical stability, low cost and easy availability of lemon peels are the additional advantages (Singh & Shukla 2016; Meseldzija *et al.* 2019; Mora *et al.* 2020; Pavithra *et al.* 2021). Pineapple is a well-liked tropical fruit around the world due to its perfect qualification, specific taste and nutritional wealth. Many pineapple crown leaves are produced by households and food industries and became plenty of available biomass waste (Astuti *et al.* 2019; Dai *et al.* 2020). Banana is one of the very extensively cultivated tropical fruits, planted over 130 countries. The 25% of the fruit is dry mass and the rest is water (Ali 2017; Vilardi *et al.* 2018). Watermelon (*Citrullus Lanatus*), melon (*Cucumis melo* L.), lemon, orange, pineapple (*Ananas comosus* L. Merryl) and banana rinds have a high content of hydroxyl and carboxyl groups (like cellulose, pectin, lignin, and hemicellulose). Thus, they can be used as a biosorbent for heavy metal adsorption from aqueous solution. Conventional heated pyrolysis, which has a long processing time and uses a large number of chemical agents, is widely used for the pretreatment of agricultural biomass. Microwave-assisted pyrolysis is an alternative energy and cost-saving method for biosorbents production, which shortens the synthesis time and heightens the efficiency (Du *et al.* 2021; Nzediegwu *et al.* 2021).

Increasing waste production and aqueous solution contamination caused by heavy metal, because of the continuous population growth, has been transformed to a global trouble. In this work, lemon, orange, watermelon, melon, pineapple and banana rinds, generally treated as waste products, were used as an alternative economic adsorbent for zinc adsorption from aqueous solution and their adsorption capacities were compared for the first time. This work aimed to get biosorbent from lemon, orange, watermelon, melon, pineapple and banana rinds by chemical activation and microwave pretreatment. Biosorbent source kind, microwave irradiation and adsorption contact time effects on zinc removal efficiency were studied. The most effective biosorbent resource between the six fruit rinds was determined, the optimum biosorbent producing conditions were decided and the product characterization was realized. In this study, biosorbent could be made in a very short period compared to conventional methods, due to the microwave pretreatment. Using the outcomes of this study, six different fruit wastes can be recycled for producing low-cost biosorbent, helping the public economy and ecological protection.

MATERIALS AND METHODS

Materials

The lemon, orange, watermelon, melon, pineapple and banana rinds investigated in this work were purchased in June 2016 from a regional greengrocer in Istanbul. The chemical activation agent, $ZnCl_2$, utilized in the study was bought from Merck (Darmstadt, Germany).

Preparation of the biosorbent

First, the lemon, orange, watermelon, melon, pineapple, and banana rinds (1 kg fruit rind for each fruit) were peeled, washed with cold tap water then by pure water and dried in an oven at 105 °C for 24 h. Next, the dried rinds were milled and sieved to less than 180 μm particle size. Finally, the produced rind dust was preserved in polyethylene bags at room temperature (22 ± 0.5 °C) (Khan *et al.* 2017; Bellahsen *et al.* 2021).

Chemical activation of biosorbent

The lemon, orange, watermelon, melon, pineapple, and banana rinds (30 g fruit rind for each fruit) were chemically activated with $ZnCl_2$ (90 g $ZnCl_2$), to increase the adsorption efficiency. The fruit rinds were mixed with $ZnCl_2$, at a $ZnCl_2$/starting material weight ratio of 3:1 (90 g $ZnCl_2$/30 g fruit rind), in a magnetic stirrer (at 500 rpm stirring speed), at 75 °C for 2 h. Activated fruit rinds were washed with distilled water until a pH of 5 was reached, and then dried at 105 °C for 18 h (Kumar & Jena 2015; Boeykens *et al.* 2019; Thompson *et al.* 2020).

The microwave pretreatment of biosorbent

The microwave pretreatment of the chemical activated and dried lemon, orange, watermelon, melon, pineapple, and banana rinds was applied utilizing a Bosch HMT72G420 microwave oven (Gerlingen, Germany) with 800 W maximal power and 2.45 GHz (wavelength 12.2 cm) operating microwave frequency. Dried rinds (1 g) were placed into 2 watch glasses and put reciprocally in the microwave oven. Microwave carbonization was applied under fixed microwave power (800 W) for 1, 2, 3, or 4 min. The resulting product was weighed, and weight loss is given in Table 1. After then, it was mixed with 0.5 M HCl in a magnetic stirrer (at 500 rpm stirring speed), at 75 °C for 1 h, washed with distilled water until the pH value of the filtrate was 5, and dried at 105 °C for 18 h (Astuti *et al.* 2019; Du *et al.* 2021; Zhou *et al.* 2021).

Table 1 | Weight loss (%) of dried peels after microwave processing (800 W; 1, 2, 3, or 4 min)

	Weight loss (%)			
Adsorbent	**800 W-1 min**	**800 W-2 min**	**800 W-3 min**	**800 W-4 min**
Lemon	3	9	17	28
Orange	4	10	22	28
Watermelon	0	6	11	27
Melon	3	7	14	30
Pineapple	2	4	9	16
Banana	4	9	12	26

Adsorbent characterization

PerkinElmer Spectrum One FT-IR spectrometer (Waltham, MA, USA) equipped with a universal attenuation total reflectance sampling accessory with a spectral range between 1,800 and 650 cm^{-1} was used for adsorbent characterization. A Perkin Elmer Lambda 35 UV-Vis Systems spectrophotometer, having a range of 190–1,000 nm, was utilized to survey the equilibrium concentration of zinc at 300 nm after adsorption (Masoudian *et al.* 2019; Thompson *et al.* 2020; Asimakopoulos *et al.* 2021).

Adsorption experiments

The conditions for zinc adsorption from aqueous solution were as follows: the stock solution was made by dissolving 40 mg/L zinc solution (50 mL) at room temperature (22 ± 0.5 °C). 0.25 g of the biosorbent produced was combined with the stock solution, at 500 rpm stirring speed and 2 or 4 h stirring time. The solution was distinct from the biosorbent utilizing filter paper and the zinc concentration was precisely determined by UV-Vis spectroscopy. All the experiments were applied three times (Li *et al.* 2019). The adsorbed zinc amount (adsorption capacity, q_e) and zinc removal yield (% *Removal*) were calculated by applying Equations (1) and (2), respectively.

$$q_e = (C_o - C_e) \times V/M \tag{1}$$

$$\%Removal = \frac{C_o - C_e}{C_o} \cdot 100 \tag{2}$$

Here, q_e is the adsorbed zinc quantity per gram of biosorbent (mg g^{-1}), C_o is the initial zinc concentration (mg/L), C_e is the unabsorbed zinc concentration in the solution (mg/L); V is the zinc solution volume (L), and M is the biosorbent quantity (g) (Hu *et al.* 2020; Saravanan *et al.* 2021).

RESULTS AND DISCUSSIONS

Characterization of the dried rinds and biosorbents

The functional groups of the dried lemon, orange, watermelon, melon, pineapple and banana rinds, and of the biosorbents made by activating the rinds with $ZnCl_2$ were determined by FT-IR spectroscopy (Figure 1). The adsorption band at about 3,276–3,393 cm^{-1} demonstrated O-H stretching vibration of hydrogen-bonded carboxylic acid, phenol and alcohols. The band at 2,894–2,920 cm^{-1} might be due to antisymmetric and symmetric stretching of C–H bond of methyl and methylene groups and aliphatic acids. The band at 1,599–1,734 cm^{-1} demonstrated carbonyl C=O groups (carboxylic acid, acetate groups, ketone, aldehyde) and the stretching vibration bond of (C=O) and (C=C). The bands at 1,008–1,038 cm^{-1} belong to C-O stretching vibrations in alcohols, phenols, ether and ester groups. Agricultural by-products are generally composed of lignin and cellulose; citrus peels are also rich in polysaccharide-pectin. They might contain proteins and polyphenols at a certain level. The functional groups of these agricultural by-products, such as alcohols, aldehydes, ketones, carboxylic, phenolic and ether groups, have the ability to bind heavy metals by donation of an electron pair from these groups to form complexes with metal ions in aqueous solution (Shakoor & Nasar 2016; Khan *et al.* 2017; Vilardi *et al.* 2018; Astuti *et al.* 2019; Masoudian *et al.* 2019; Meseldzija *et al.* 2019; Safari *et al.* 2019; Shakya & Agarwal 2019; Yusuf *et al.* 2020). The interaction of the functional groups present on the surface of the biosorbents deteriorates the lignocellulosic structure of the fruit rinds. FT-IR spectra of the fruit rinds and the obtained biosorbents showed that the structure of the fruit's rinds had altered, and some peaks had been lost (Romero-Cano *et al.* 2017).

Adsorption analysis results

Waste fruit rinds were used as biosorbent for zinc adsorption from aqueous solution, after microwave pretreatment and chemical activation with $ZnCl_2$.The produced biosorbent was mixed with a zinc stock solution, which was prepared from 40 mg/L standard zinc solution. The adsorption experiments were realized with 50 ml of treatment solution at room temperature (22 ± 0.5 °C). The fixed biosorbent dose was 0.25 g/L, the stirring speed was 500 rpm and stirring time was 2 or 4 h. UV-Vis spectroscopy was used to determine the zinc concentration in the solution, the results were calculated in mg/L and are given in Figures 2 and 3. All the experiments were applied three times.

UV-Vis analysis results of microwave pretreated and $ZnCl_2$ activated biosorbents showed that maximum zinc removal yield of the lemon rind was 97.95%, orange rind was 89.45%, watermelon rind was 36.90%, melon rind was 32.65%, pineapple rind was 30.50% and banana rind was 22.50%. The zinc removal yields obtained using lemon and orange rinds were higher than those reported from studies with green marine macro alga *Caulerpa scalpelliformis* (83.3%) (Jayakumar *et al.* 2021), Scots pine (*Pinus sylvestris* L.) and Silver birch (*Betula pendula*) (35–37%) (Komkiene & Baltrenaite 2016); sunflower, potato, canola and walnut shell residues (43.2%) (Feizi & Jalali 2015); almond shell (80%) (Çoruh *et al.* 2014); tea waste (79.5%) (Wen *et al.* 2017). UV-Vis analysis results demonstrated that the zinc adsorption efficiency increased with the increase of microwave pretreatment time. Since the biosorbent surface was unsaturated in the beginning, zinc adsorption took place rapidly. With increasing contact time, the adsorbent surface gradually saturated and the adsorption capability increased till equilibrium was achieved. Development of the adsorption capacity is because of the introduction of carboxyl groups on the adsorbent surface during their preparation, which are involved in the adsorption of metal

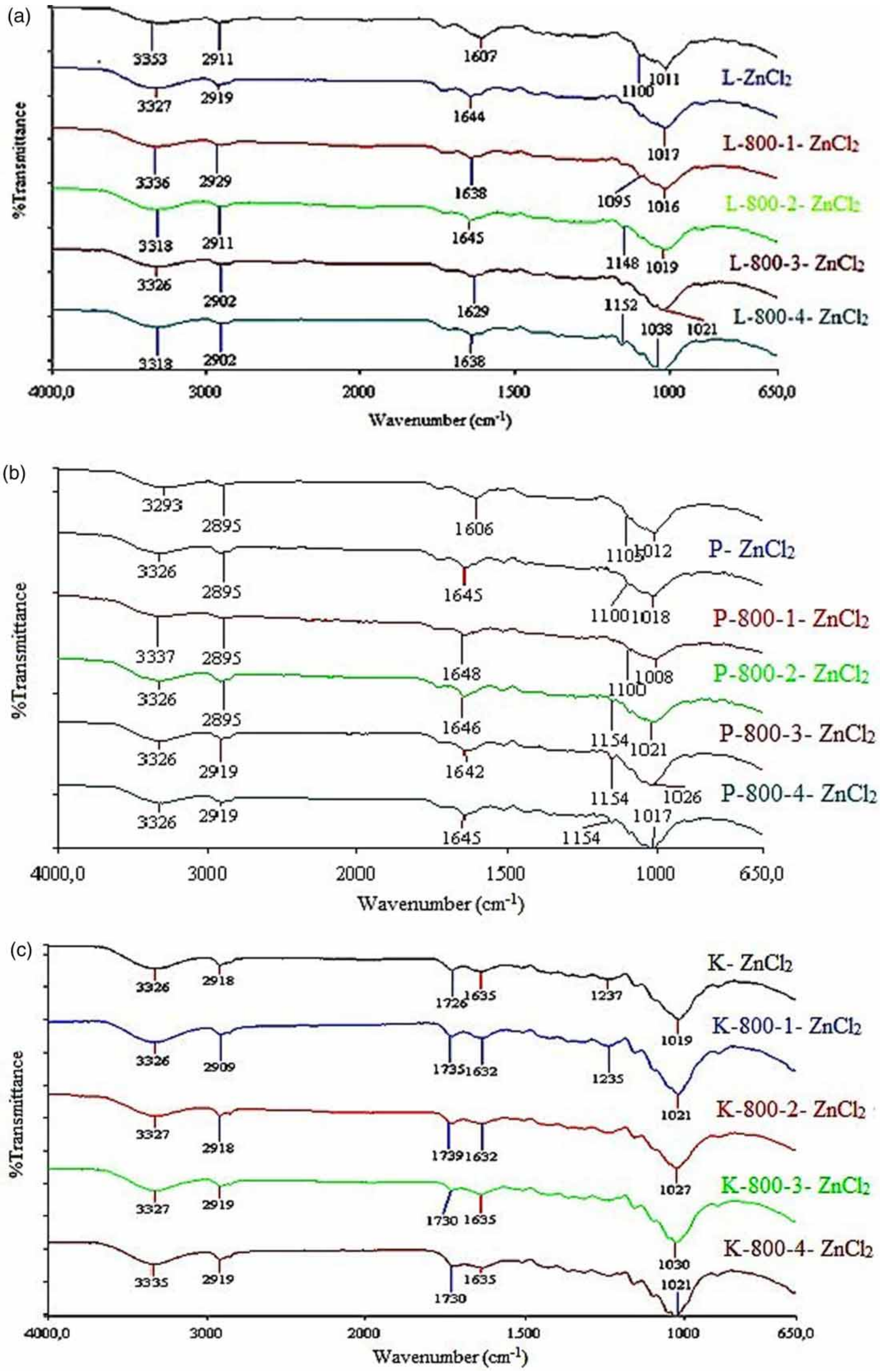


Figure 1 | FT-IR spectra of the dried (105 °C, 24 h.) rinds, and of the rinds activated with $ZnCl_2$ and microwaved (800 W) for 1, 2, 3, or 4 min. (a) Lemon, (b) orange, (c) watermelon, (d) melon, (e) pineapple, (f) banana. (*continued.*)

(Romero-Cano *et al.* 2017; Ngabura *et al.* 2018). Optimum results for the six peels were acquired with lemon and orange rinds left in the microwave at 800 W for 4 minutes.

CONCLUSIONS

In this study, lemon, orange, watermelon, melon, pineapple and banana rinds were chemical activated with $ZnCl_2$ ($ZnCl_2$/fruit rind weight ratio of 3:1) and carbonized by microwave (1, 2, 3 or 4 min. at 800 W microwave

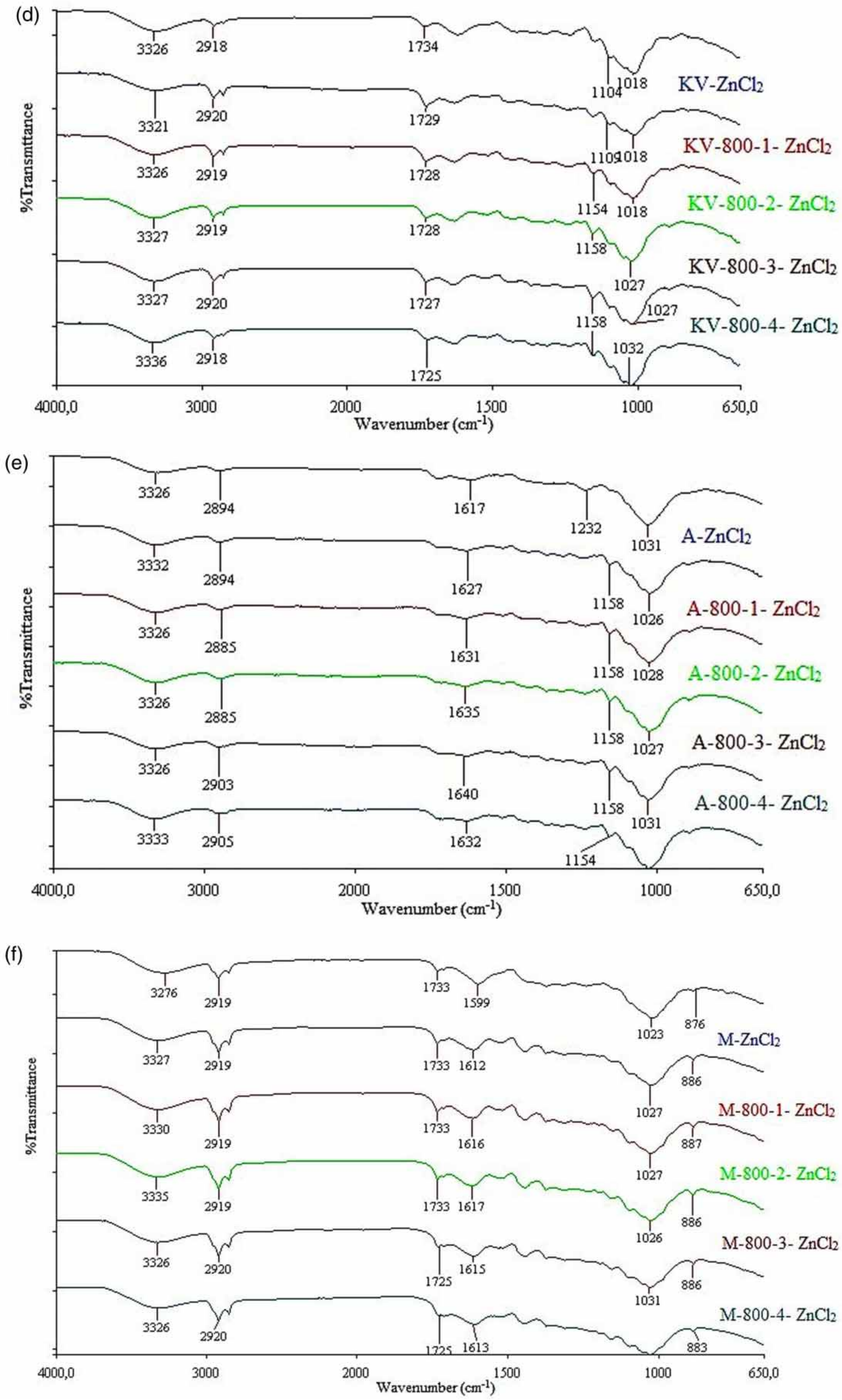


Figure 1 | Continued.

power) to produce an economical biosorbent for zinc adsorption from aqueous solution and their adsorption yields were compared. The characterization of the dried rinds and synthesized biosorbents was performed by FT-IR spectroscopy, which was utilized to determine the functional groups of the dried lemon, orange, watermelon, melon, pineapple, and banana rinds, and of the biosorbents obtained by activating the rinds with $ZnCl_2$. The

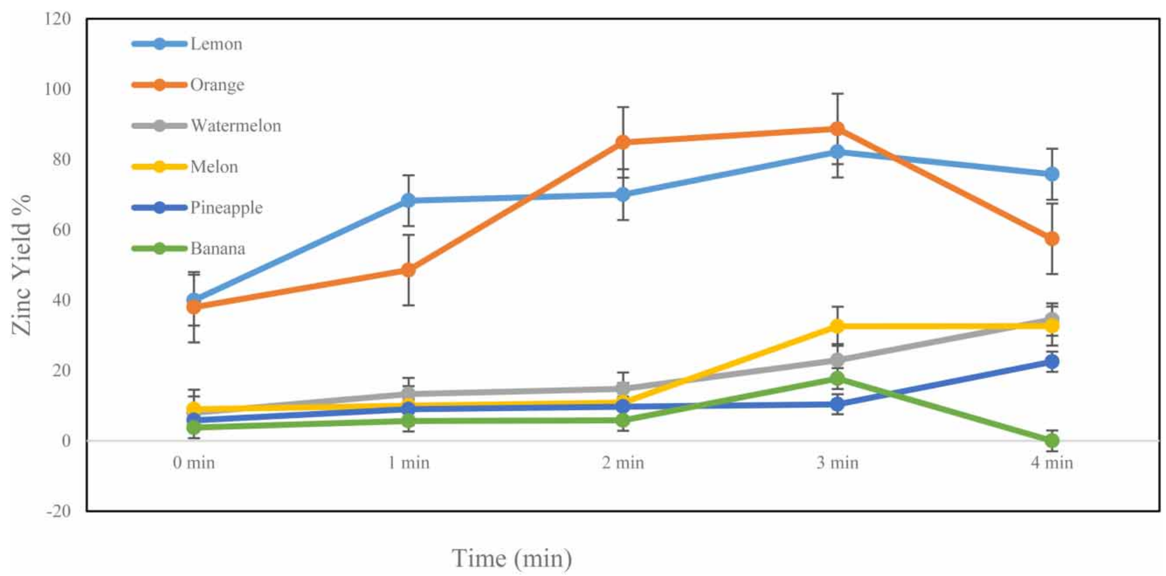


Figure 2 | Microwave pretreatment time (1, 2, 3, or 4 min.) effects on Zinc adsorption yield (2 h).

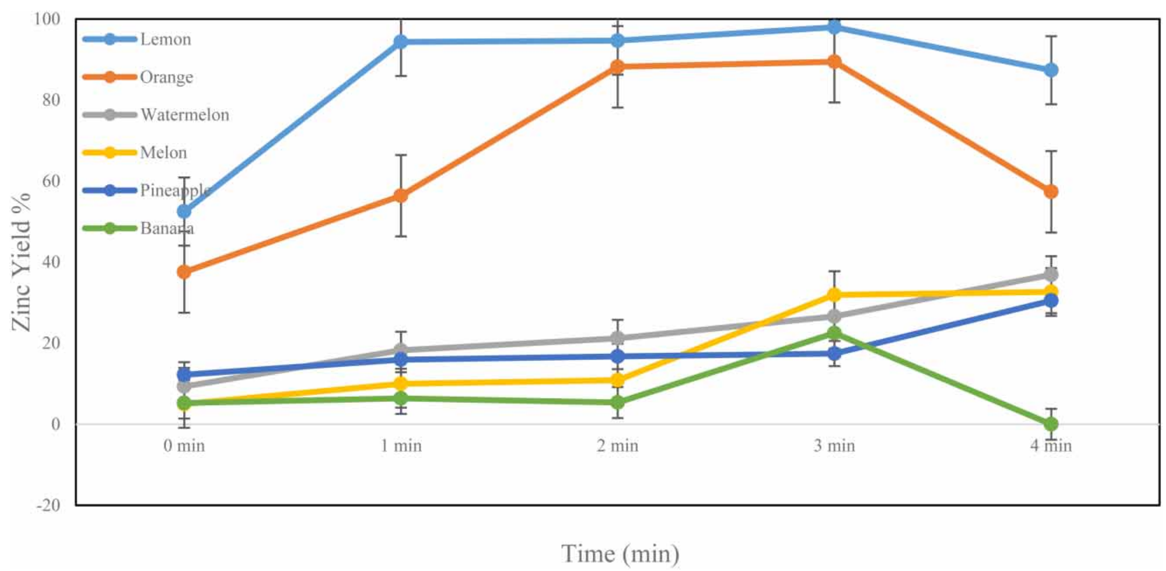


Figure 3 | Microwave pretreatment time (1, 2, 3, or 4 min.) effects on Zinc adsorption yield (4 h).

interaction of the functional groups present on the surface of the biosorbents deteriorate the lignocellulosic structure of the fruit rinds. Because of this, the FT-IR spectra of the fruit rinds and obtained biosorbents showed that the structure of the fruit's rinds had altered, and some peaks had been lost. Zinc biosorption yield of the synthesized biosorbents was determined using UV spectrophotometer. The produced biosorbent was mixed with a zinc stock solution, which was prepared from standard zinc solution with a concentration of 40 mg/L. The zinc concentration was decided by UV-Vis spectroscopy. UV-Vis analysis results of microwave pretreated and $ZnCl_2$ activated biosorbents showed that the highest zinc adsorption yield of the lemon rind was 97.95%, orange rind was 89.45%, watermelon rind was 36.90%, melon rind was 32.65%, pineapple rind was 30.50% and banana rind was 22.50%. The best results for the 6 peels were obtained with lemon and orange peels left in the microwave at 800 W for 4 minutes. Maximum zinc biosorption capability was determined 39.18 mg/g for lemon, 35.78 mg/g for orange, 14.76 mg/g for watermelon, 13.06 mg/g for melon, 12.2 mg/g for pineapple and 9 mg/g for banana rinds. The outcomes of this study showed that lemon, orange, watermelon, melon, pineapple and banana rinds, being natural, economical, eco-friendly, having high biosorption capability and rate, can be utilized as a successful biosorbent for the treatment of aqueous solutions including zinc metal ions.

ACKNOWLEDGEMENT

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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