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Precipitation of Solid Waste in Olive Mill Wastewater by Coagulation using Calcium Carbonate ($CaCO_3$)

Khaled Rawajfeh*[1)], Arwa Sandouqa[1)], Aiman Al-Rawajfeh[2)]

[1)]*The University of Jordan, School of Engineering, Chemical Engineering Department, Amman, 11942, Jordan.*
[2)] *Tafila Technical University, Department of Chemical Engineering, 66110, Jordan.*

Abstract

This study aimed to use a chemical coagulant to treat Olive Mill Wastewater (OMW) in such a way as to improve and expedite the process of precipitating the solid waste material present in the wastewater. This process is of extreme importance in harvesting the solid material that can be used as domestic or industrial fuel material and as a supplement to animal feed. The chemical coagulant used in this study was calcium carbonate ($CaCO_3$). The effect of the addition of this coagulant on some physical properties of olive mill wastewater (OMW) such as pH, electrical conductance (EC), the precipitated amount of the total suspended solids (TSS), and the higher heating value (HHV) of the precipitated solids was studied. It was found that small amounts of $CaCO_3$ need to be added to affect the operation. The study results showed that the optimum value to be used was around three wt/wt %.

Paper type: Research paper

Keywords: Coagulation, calcium carbonate, decantation, mixing, centrifuge, solid waste, olive mill wastewater, evaporation, drying, olive oil.

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Introduction

Olive mill wastes cause significant environmental problems, especially in Mediterranean areas where they are generated in huge quantities in short periods. In Jordan, olive mills (136 mills with a total capacity of about 947,592 tons/year) generate olive cake which can be processed as pressed blocks (called *Jift*). The olive cake is considered one of the essential traditional domestic fuel sources in rural areas, supplementary animal feed, and industrial fuel material (Al-Hmoud *et al.*, 2020). Currently, the OMW is discarded in open land ditches, causing severe environmental pollution that affects groundwater quality. Each pressed ton of olive generates about 1.5 m^3 of dark red to black effluent called olive mill wastewater (OMW) which contains phenols, free fatty acid, high biological oxygen demand (BOD), and high chemical oxygen demand (COD). When the OMW is disposed of in open-air evaporation ponds, most of the water evaporates. The solid waste material present in the OMW (Olive Mill Solid Waste OMSW) precipitates in the pond's bottom (Lakhtar *et al*., 2009). The total amount of olive cake produced reached about 30,000tonnes/year in 2017 with about 40,680 m^3 of OMW (Otles and Selek, 2012; Adhoum and Monser, 2004; Ministry of agriculture's annual report, 2019; Al-Enazi *et al*., 2013). The olive oil extraction process results in clean olive oil, solid waste (cake), and OMW containing fine solid particles. Most of the OMW is produced mainly in a three-phase process, as shown in **Figure 1**. In the three-phase process, the first stage of the separation process is the decanter (a solid bowl centrifuge), which issues three products. The olive cake is slightly moist, containing small amounts of oil (less than 5%).

* Corresponding author: E-mail: k_rawajfeh@ju.edu.jo ORCID: https://orcid.org/0000-0002-4618-5122
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Screw conveyors outside the mill house discard this product off. Two liquids leave the decanter: the main olive oil stream containing small amounts of wastewater; and the main OMW containing small amounts of olive oil (less than 1%). These two liquid streams are sent to the second stage centrifugation processes to extract the olive oil from the olive oil-rich liquid stream; the other liquid stream is sent to another centrifuge to recover the small amounts of oil in it. All the wastewater from both stages is sent to storage, ready for disposal.

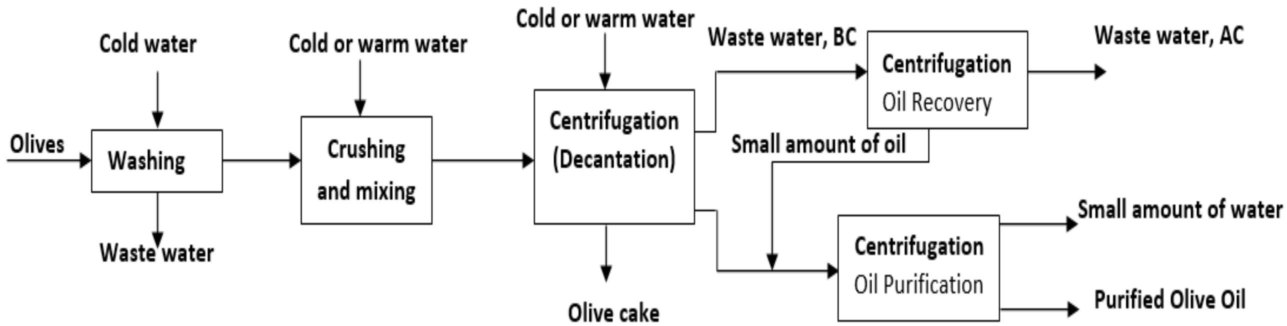

Fig. 1 A three-phase extraction process.

The disposal of untreated OMW into open lands is considered a crucial ecological problem that deteriorates the environment in Jordan. Due to the great variety of compounds in the waste, such as long-chain fatty acids and phenols, several technologies to remove the harmful compounds for the protection of the environment are used for this purpose. Many scientists worked on specialized alternatives processes to treat OMW such as physical (evaporation, centrifugation), chemical (coagulation/flocculation), biological (aerobic and anaerobic treatment), and advanced methods (ozonation: Fenton process) (Otles, and Selek, 2012; Montero *et al*., 2015; Shaheen and Karim, 2007). The chemical treatment by coagulation is a pretreatment process to remove suspended solids before further liquid treatment. Azraq bentonite, reddish volcanic tuff, lime, aluminum sulfate, ferric chloride, and sodium carbonate were investigated by Gharaibeh for the removal of turbidity and COD from OMW (Gharaibeh *et al.*, 2008). Results of using different agents to remove the turbidity measured in Nephelometric Turbidity Units (NTU) and the Chemical Oxygen Demand (COD) are given in **Table 1**.

The use of Alum was not recommended because of its cost. The treated OMW is not recommended to be used for irrigation due to its high dissolved solid content and toxic chemicals, such as phenols, polyphenols, humic acids, and lipids (Al-Hmoud *et al.*, 2020; Gharaibeh *et al.*, 2008).

Table 1 NTU and COD removal from different coagulants (Gharaibeh *et al*., 2008).

Agent	NTU % removal	COD %removal
Alum	95	65
Lime	99	69
Azraq bentonite	96	37.5
Reddish volcanic tuff	Not efficient	Not efficient
Ferric chloride	Not efficient	Not efficient
Sodium carbonate	Not efficient	Not efficient

Meyssami and Kasaeian, used starch, Alum, chitosan, and ferric chloride as chemical coagulants, followed by flotation to remove oil droplets from suspension (OMW), and showed that ferric chloride and starch were not effective coagulants. In contrast, Alum and chitosan were effective in reducing the turbidity of the emulsions by (90%). In the optimum coagulation and flotation stages (100 ppm chitosan, airflow 3L/min, aeration time 45sec, 20°C, pH 6), the COD reduction reached up to 95% (Kilic and Solmaz, 2013; Meyssami and Kasaeian, 2005).

Cationic and anionic poly-electrolytes were tested on OMW treatment by Sarika *et al*., lime and ferric chloride were used as reference coagulants, and the results compared with the poly-electrolytes. Most of the poly-electrolytes were found to remove total suspended solids (TSS), COD, and BOD_5. The results showed that lime and ferric chloride are pretty effective in TSS removal, although the degree of COD reduction was generally lower than that with poly-electrolytes (Sarika *et al*., 2005).

Various inorganic chemicals and organic poly-electrolytes were investigated by Ginos *et al*. Inorganic chemicals, such as iron, lime, magnesium, and aluminum, and cationic and anionic commercial poly-electrolytes were added either alone or in various combinations and evaluated the removal efficiency of TSS and COD. The combination between lime and ferrous sulfate with cationic poly-electrolytes showed about 10-40% removal efficiency of COD and 30-80% total phenol (Ginos *et al*., 2006).

The current study examined a novel approach for producing olive cake from olive mill wastewater (OMW) using a combination of coagulation-sedimentation and evaporation processes. The sedimentation rate of the fine solid material is enhanced by the addition of appropriate amounts of Ca-based coagulant, namely calcium carbonate ($CaCO_3$), which accelerates the rate of precipitation and maximizes the amount of precipitated fine solid material.

1 Materials and Methods

1.1 Olive mill wastewater

Olive Mill Wastewater (OMW) collected from a three-phase olive mill located in the South of Jordan was used in this study. Two sources of the OMW were studied. One produced in the decantation process just before the oil recuperation centrifuge (designated as BC: Before centrifuge), while the other after the separation centrifuge recuperates the small amounts of oil present in the OMW issuing from the decanter (designated as AC: After centrifuge). The chemical coagulant used in this study was calcium carbonate ($CaCO_3$). The efficiency of the treatment resulting from the use of the coagulant was evaluated by measuring EC, pH, and TSS of OMW samples according to the Standard Methods for the Examination of Water and Wastewater (American Public Health Association, 2005). The HHV for the precipitated solids was determined as indicated previously. All measurements on these parameters and experiments on oil extraction and HHV were conducted on samples obtained after sedimentation. All procedures are explained in the following sections.

1.2 Methodology

1.2.1. Olive mill wastewater characterization

The physical analysis covered the measurement of pH, total suspended solids (TSS), electrical conductivity (EC), oil content, and the higher heating value (HHV) of the solid presence in the OMW. The analyses were carried out according to the American Public Health Association for Examination of Water and Wastewater APHA (2005). pH and EC measurements were carried out using calibrated multi dual pH/EC meter (Mettler S47). The high heating values (HHV) were directly determined using a bomb calorimeter (model 6400 series manufactured by Parr Instrument Company). The HHV includes the latent heat of the vapor emitted from the specimen.

1.2.2 Oil extraction

The oil content was quantified using a Soxhlet extraction apparatus. The olive oil in the olive cake obtained from the OMW was extracted by hexane. Extraction was continued until no additional color was leached from the sample with 15 cycles (Cordova *et al*., 1998).

1.2.3 Coagulation-Sedimentation

Removing suspended solids from wastewater by gravity separation is one of the most widely used unit operations in wastewater treatment (Aly *et al*., 2014). In this study, physical separation was carried out using centrifuge tubes shown in **Figure 2**, in the presence of the coagulant or without. These tubes are made of polyethylene, having dimensions as shown in the figure. The tube is filled with the suspended material to a height of 7.5 cm above the conical bottom. The tests were carried out at different temperatures 15, 25, and 35°C with different doses of coagulant 0.5, 1, 1.5, 2, 2.5, and 3% (wt/wt). In order to determine the optimum dosage of the coagulant, the required dose was mixed with 45 g of OMW sample for 1 min. The resulting sludge was allowed to settle, and the settling rate was measured in terms of the change of the height of the interface of the clear solution at the top of the tube with time.

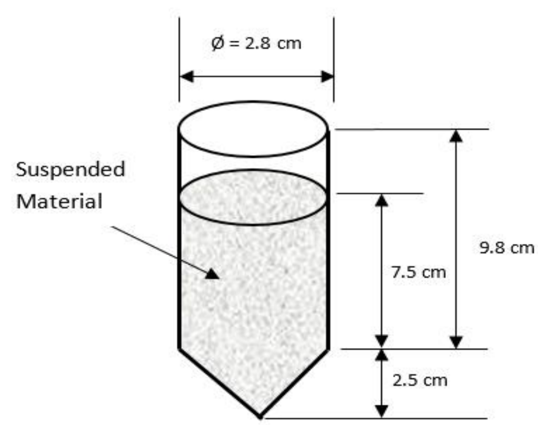

Fig. 2 Dimensions of the sedimentation tubes.

1.2.4 Drying rate

Considering the climate conditions in Jordan: such as aridity, high solar energy, and wind velocity, natural evaporation of treated OMW in ponds might be an acceptable solution. The natural evaporation is enhanced by treating the OMW first with the chemical coagulant.

A tray drier was used to evaluate the drying rate of the treated OMW. The apparatus consists of an air duct mounted on a floor-standing frame. Air is blown into the duct by a fan whose speed can be controlled. The air passes over an electrically-heated element fixed in the central section of the duct, where trays of the OMW to be dried are placed in the air stream. The trays are carried on a support frame attached to a digital balance mounted above the duct. Air is discharged to the atmosphere through an outlet duct section in which a digital anemometer is mounted to measure air velocity (1.86m/s). Wet and dry bulb temperatures of the air are measured using an aspirated psychrometer. Two kilograms of each type of sample were uniformly spread on a tray (surface area=0.06m^2). Variables such as weight loss, dry and wet air temperatures were monitored regularly.

2 Results and Discussion

The measured physicochemical characteristics of OMW samples used in this study are given in **Table 2**. It is seen that there are slight differences between the results of the two types of samples. This is because the centrifugation of the OMW issuing from the decanter mainly removes some of the small amounts of oil in it. This oil is sent back to the main oil stream. Total suspended solids concentrations ranged from 113.2 to 124.6g/L; most of them are colloidal, making their settling ability very difficult (Al-Enazi *et al*., 2013). The EC values of OMW samples were around 8 and 11mS cm^{-1} for (AC) and (BC) samples, respectively. This parameter indicates the high mineral contents (S'habou *et al*., 2009). The percentage of dry matter of (BC) samples was higher than those of (AC). Moreover, a notable difference was observed in the oil content between the two sludges obtained from these two sources of samples, which corresponds to the centrifugation effect. These results are following available literature results (Gharaibeh *et al*., 2008).

Table 2 Physicochemical characteristics of OMW samples.

Parameter	Before Oil recuperation centrifuge (BC)	After Oil recuperation centrifuge(AC)
pH	4.95	4.79
EC (ms/cm)	11.01	8.24
Water % wt	89.6	92.17
HHV (cal/g)	6720	6200
Oil content (% wt)	34.7	28.9
TSS (g/l)	124.6	113.2

2.2 Coagulation-Sedimentation

2.2.1. Sedimentation

Sedimentation experiments were conducted to determine the amount of the settleable solids in the OMW using centrifuge tubes before adding coagulants. The results showed that the settled solid fraction for both types of samples (BC, AC) was negligible, even after a very long sedimentation time. This may be due to high TSS concentrations, making settling very difficult (S'habou *et al*., 2009).

2.2.2. Effect of coagulant's dosage on sedimentation rate

The effect of the addition of $CaCO_3$ and its dosage on sedimentation rate as investigated was significant, as shown in **Figure 3** when the dose of $CaCO_3$ was varied from 0.5 to 3wt/wt%. It was seen that after a given time, the height of the clear solution increased significantly with the increase of the dose of the coagulant. The optimum dose for $CaCO_3$ was found to be around 3 (wt/wt) % at contact times of 4 and 6 minutes for BC and AC samples, respectively. The highest clearance (4cm) was achieved by using 3wt/wt % dosage after 6 minutes for (AC) samples, while the maximum clearance in (BC) samples was (3cm) after 4 minutes. This may be due to the higher oil content in (BC) samples, which decreases the sedimentation rate, and accordingly, the sludge removal will decrease.

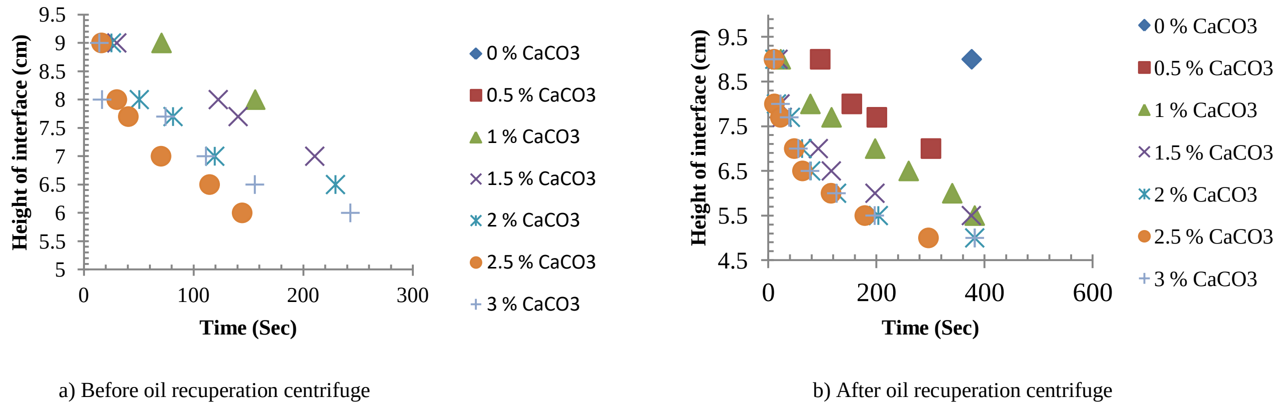

Fig. 3 Effect of $CaCO_3$ dosage on sedimentation rate (percentages are wt/wt).

These results demonstrate clearly that chemical coagulation by $CaCO_3$ effectively reduces the amount of sludge in OMW in a shorter time.

2.3. Total suspended solids (TSS)

Total suspended solids (TTS) concentrations of OMW ranged from 113.2 to 124.60g/L. Most of the TSS are colloidal, making the samples settling rate prolonged. The precipitation of the suspended solids by using $CaCO_3$ was 91.1, 94.2% for BC and AC samples, respectively as shown in **Figure 4**. That was in good agreement with previously described results by using poly-electrolyte obtained by Sarika (Sarika *et al*., 2005), and much better than lime treatment which gives about 53.2% removal (Aktas *et al*., 2001).

2.4. Effect of coagulant dose on pH

The initial pH of BC and AC olive mill wastewater used in this study were 4.95 and 4.79, respectively. The pH of OMW increased after treatment with $CaCO_3$ to about 6, as shown in **Figure 5**. This change in pH may be due to the neutralization of some of the phenols upon adding $CaCO_3$ to the OMW. Phenols are acidic in solution; hence their neutralization is thought to reduce the solution's acidity, increasing the pH (Azzam *et al*., 2010). The increase of pH after treatment demonstrates the substantial ability of the $CaCO_3$ to neutralize OMW by increasing the hydrogen ion concentration, so the negative surface charge of the suspended hydrophilic colloids is reduced, leading to neutralization acidity (Niaounakis and Halvadakis, 2006; Lagoudianaki, 2003).
It is noted that the effect of the addition of the coagulant on the pH of both types of samples is almost the same and their values are almost the same. This means that the neutralization affects the OMW regardless of the presence of small amounts of oil or not.

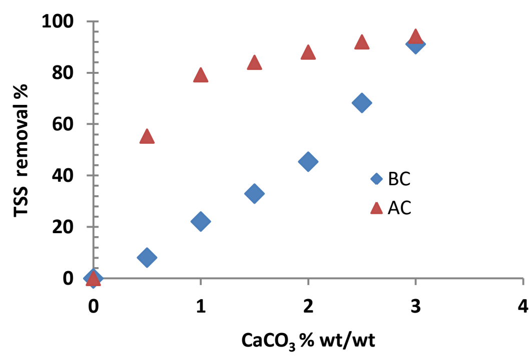

Fig. 4 Effect of coagulant dose on the removal of TSS.

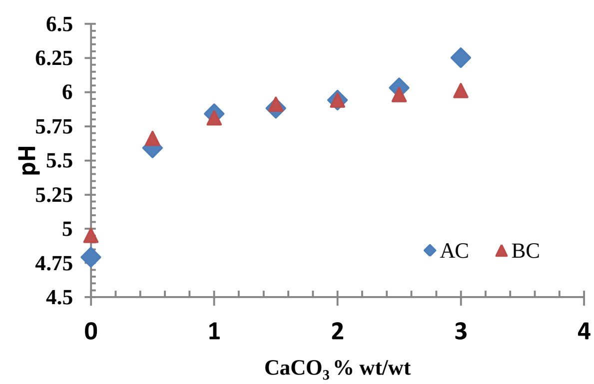

Fig. 5 Effect of coagulant dose on pH.

2.5. Effect of coagulant dose on electrical conductivity (EC)

The effect of the addition of the coagulant on the EC is shown in **Figure 6**. The EC Increased after coagulation by 32% and 48% for BC and AC samples, respectively, and then it remained almost the same regardless of the amount of coagulant added to the OMW. The reasonable explanation behind the increase in EC values by increasing the coagulant dose can be attributed to the formation of Ca^{+2} and CO_3^{-2} ions that increased the EC (S'habou *et al.*, 2009). It is noticed that the difference in EC between the BC and AC samples remains almost the same even after treatment with the coagulant.

2.6. Effect of coagulant dose on olive cake heating value

Olive cake can be used as an auxiliary solid fuel in the industry using its heat content. Heating value is one of the core determinants of the suitability of sludge as a source of solid fuel (Saidur *et al.*, 2011). Olive cake samples obtained from BC and AC are characterized by an average heating value of 6200 and 6720cal/g. It can be seen from **Figure 7** that the addition of $CaCO_3$ decreases the calorific values of both types of samples. The decrease in calorific value would not be acceptable since the whole aim of biomass combustion is to enhance its healing values. Therefore, it is essential to consider the effect of coagulant to be added in the solid biofuel preparation (Zakari *et al.*, 2013). However, the calorific values are observed to decrease by not more than about 3% for each sample by adding 3%wt/wt of coagulant when compared with the values of the olive cake without adding coagulant. It is noticed that the HHV of the cake obtained from BC samples is higher than that obtained from AC samples. The difference is attributed to the presence of olive oil in the cake obtained from the BC samples. The difference remains the same regardless of the amount of added coagulant.

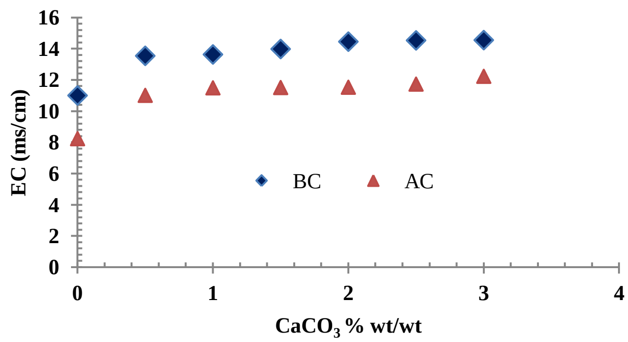

Fig. 6 Effect of coagulant dose on EC.

2.7. Drying rate

Drying is a fundamental stage for waste revaluation as biomass. High moisture content implies that some heat from combustion evaporates water in biomass, reducing the valuable energy. Lower moisture allows a higher flame temperature (with better temperature gradients and heat transfer), as well as shorter residence time in the combustion chamber (Montero *et al.*, 2015).

Drying rates were calculated according to equation (1):

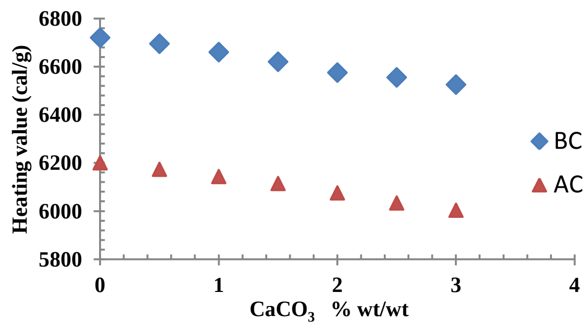

Fig. 7 Effect of coagulant dose on heating value

$$\mathbf{R} = \frac{S}{A}\frac{dX}{dt} \quad (1)$$

Where, R=drying rate (kg H_2O/min.m^2), *X*=moisture content on dry basis (kg H_2O/Kg dry solid), *A*=tray surface area, m^2, *t*=time, min, *S*=weight of dry solid, (kg).

The experimental data and the calculated evaporation rates for both types of samples are given in **Tables 3** and **4**. Moreover, the rates of evaporation versus time are shown in **Figures 8** and **9**. The overall mean evaporation rate for both types of samples was calculated based on data present in **Tables 3** and **4**, and it was found to be identical at a value of 6.5 kg H_2O/m^2 d based on an 8-hour working day. This result is comparable to literature values obtained by Galliou *et al.*, (Galliou *et al.*, 2018), who reported a value of 5.2 (kg H_2O/m^2.d) (Galliou *et al.*, 2015). The results, as seen in Figs. 8 and 9 indicate two stages of drying, which can be thought of as evaporation of free moisture and bound moisture content. Between those two stages, a somewhat slower rate of drying is observed. It is seen that the reduction in water content was higher for AC samples than for BC samples. The moisture content (*X* as defined previously) was reduced from 0.95 to 0.08 for AC samples and 0.84 to 0.1 for BC samples. The discrepancy is due to the presence of oil in the BC samples. The drying rate in both stages for both types of samples is linear. The rates for both stages in AC samples (0.0059 and 0.0031) are higher those for BC samples (0.0047 and 0.0026). In addition to that, the drying rates are higher in the first stage evaporation for both types of samples. This consolidates the idea of the existence of the two types of evaporation. The rate of evaporation in either of the first stages of evaporation is faster than in the second.

Table 3 Evaporation results for AC samples.

Time, t (min)	Evaporated H_2O (kg)	Moisture Dry Basis, X (kg Water/ kg Dry Solid)	Drying rate, R (kg H_2O/min m^2)
2	0.148	0.95	0.000
4	0.145	0.93	0.025
6	0.143	0.91	0.017
8	0.14	0.89	0.025
10	0.138	0.88	0.017
15	0.134	0.86	0.013
20	0.13	0.83	0.013
25	0.125	0.80	0.017
30	0.12	0.77	0.017
35	0.115	0.73	0.017
40	0.11	0.70	0.017
45	0.099	0.63	0.037
50	0.096	0.61	0.010
60	0.087	0.56	0.015
70	0.083	0.53	0.007
80	0.077	0.49	0.010
90	0.07	0.45	0.012
120	0.066	0.42	0.002
150	0.06	0.38	0.003
170	0.05	0.32	0.008
190	0.046	0.29	0.003
210	0.034	0.22	0.010
230	0.02	0.13	0.012
250	0.012	0.08	0.007

Table 4 Evaporation results for BC samples.

Time, t (min)	Evaporated H_2O (kg)	Moisture Dry Basis, X (kg Water/ kg Dry Solid)	Drying rate, R (kg H_2O/min m^2)
2	0.175	0.84	0.000
4	0.174	0.84	0.008
6	0.172	0.83	0.017
8	0.169	0.81	0.025
10	0.167	0.80	0.017
15	0.158	0.76	0.030
20	0.151	0.73	0.023
25	0.145	0.70	0.020
30	0.14	0.67	0.017
35	0.135	0.65	0.017
40	0.127	0.61	0.027
45	0.125	0.60	0.007
50	0.117	0.56	0.027
60	0.115	0.55	0.003
70	0.105	0.51	0.017
80	0.1	0.48	0.008
90	0.093	0.45	0.012
120	0.085	0.41	0.004
150	0.071	0.34	0.008
170	0.062	0.30	0.008
190	0.043	0.21	0.016
210	0.028	0.13	0.013
230	0.026	0.13	0.002
250	0.02	0.10	0.005

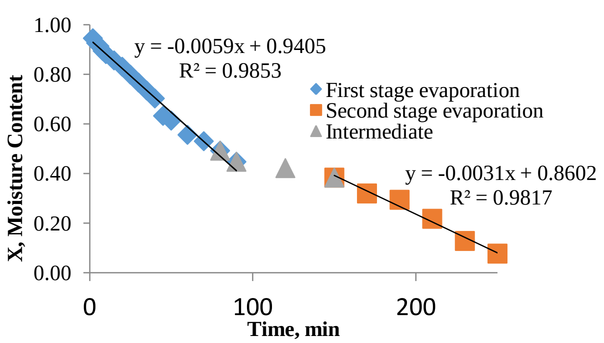

Fig. 8 Drying Rate Curves for AC Samples.

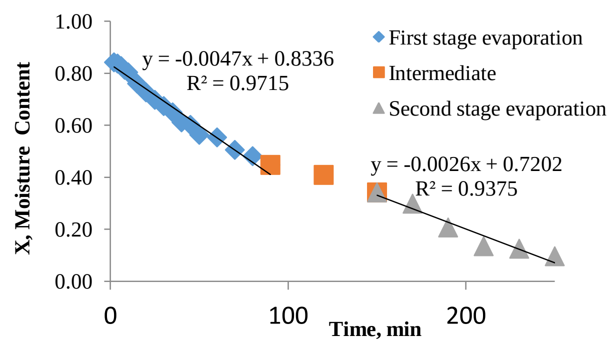

Fig. 9 Drying Rate Curves for BC Samples.

Conclusions

The main aim of this study was to treat the OMW in terms of separating the solid contents in a manner that would accelerate the separation and enhance it. The operation was achieved by the addition of a coagulant ($CaCO_3$) to the OMW while delivering this water into disposal ponds having an impermeable lower surface (cement based for example) in which the solids content will precipitate quickly allowing the remaining OMW to overflow to another pond in which it will eventually evaporate or might be reused for other purposes. The removal of TSS is almost the same from both types of samples; however, the removal from AC samples is much more effective and requires less $CaCO_3$ dosage concentration. This confirms the importance of recuperating the small amounts of oil present in the BC samples both from economic point of view of improving the TSS removal at lower coagulant concentrations. It is recommended to conduct research on a pilot project to investigate the economic implementation of the findings of this research in Jordan and to assess the possible use of the treated OMW for agricultural purposes.

Nomenclature

BOD	=Biological Oxygen Demand	[g/l]
COD	=Chemical Oxygen Demand	[g/l]
OMSW	=Olive Mill Solid Waste	[-]
NTU	=Nephlometric Turbidity Units	[NTU]
pH	=potential of Hydrogen	[-]
TSS	=Total Suspended Solids	[g/l]
$CaCO_3$	=Calcium Carbonate	[-]
EC	=Electrical Conductivity	[ms/cm]
HHV	=Higher Heating Value	[cal/g]
R	=drying rate	[kg H_2O/min m^2]
X	=moisture content on dry basis	[kg H_2O/Kg dry solid]
A	=tray surface area	[m^2]
t	=time	[min]
S	=weight of dry solid	[kg]

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