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# Removal of Dimethoate, Carbendazim and Chlorpyrifos Residues from Date Fruits and Evaluation of the Treatments Effect on The Nutrition Value

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**Abstract:** *The efficiency of several wash treatments for removing dimethoate, carbendazim, and chlorpyrifos residues from date fruits treated with pesticide solution at a concentration of 20 µg/mL was measured and optimized. Pesticide samples were extracted using the QuEChERS acetate buffered technique and then analysed using HPLC coupled with a diode array detector (DAD). The results indicated that dimethoate and chlorpyrifos clearance rates rose in the sequence of NaHCO3 >acetic acid > tap water. Additionally, the percentage of pesticide residues removed from date fruits varied according to the concentration of the washing solution, the contact time, and the temperature. However, removal rate of carbendazim was the lowest throughout all the treatments. The effect of different washing procedures on the nutritional value of date fruits revealed that all treatments had a negative impact on the total sugars content, total phenolic content, and DPPH inhibition activity. Whereas tap water at 37°C resulted in significant reductions in total phenolic content across all contact periods examined as compared to control. Date fruit antioxidant capacity decreased in all washing treatments when contact periods were 5 or 10 minutes.*

**Keywords:** pesticides, washing effect, nutrition value

## 1. Introduction

In date fruits, pesticides such as dimethoate, carbendazim, and chlorpyriphos are commonly used (El-Saeid., 2010). Many organophosphates are effective nerve agents because they inhibit acetylcholinesterase (AChE) in nerve cells. Breathing, ingestion, and dermal absorption are all possible pathways for OPs absorption. However, their toxicity is not limited to the acute stage, and its long-term effects have long been recorded. Many OPs have neurotoxic effects on developing species, even at low levels of exposure, and neurotransmitters like acetylcholine (which is impaired by organophosphate pesticides) are crucial in brain growth (Satpathy et.al., 2012).

It is necessary to estimate the level of pesticide exposure at the point of consumption to estimate the possible pesticide exposure from contaminated food. Commercial and household processing methods such as washing, peeling, blanching, and frying, have already been identified to minimize

residue levels in food, thereby reducing the effect on human health (Zhang et al., 2007, Satpathy et al., 2012). Food protection and safety is gaining increasing concern around the world due to its direct impact on human health.

Date palm fruit is common in the diets in many regions, including the Middle East and North Africa and is popularly dried or used as a condiment because of its high carbohydrate, protein, and minerals content (Considine 1982; Al-Showiman and Fayadh1990 and Osman et al.,2014). Beside of being food, the date has been used as a sugar source due to its high carbohydrate content (70–80 percent). Some antioxidants, such as protocatechuic, p. hydroxy benzoic, coumaric, and ferulic acid found in date fruits may play an important role in total antioxidant activity and contribute to human health (Besbes et al.2009). Additionally, concentrated date juices are rich of tannins and ascorbic acid (Kulkarni et al. 2010).

## 2. Literature Review

An extensive analysis of the literature shows that food processing usually results in significant reductions in pesticide residue levels in the prepared food, especially through washing, peeling, and cooking operations (Soliman, 2001; Zohair, 2001; Osman.et al.,2014).

There are some chemicals usually employed as food additives or for hygienic purposes, these products may be chosen for washing treatments such as acetic acid, hydrogen peroxide ($H_2O_2$) which is an oxidant usually used for the treatment of waters, control of excess chlorine content, and the removal of residual ozone from treated waters (Pugliese et al. 2004). Potassium permanganate is an antibacterial and disinfectant and additionally it used to treat drinking water (Osman., et al., 2014).

Because of high consuming rate of date fruits in Islamic world, there is a need to seek new methods to improve the safety of this product. Therefore, the present study was carried out to assess the efficiency of some washing solutions such as tap water ($H_2O$), acetic acid (AA) and sodium bicarbonate ($NaHCO_3$) as wash treatments for removal of three common pesticides in date fruits namely, dimethoate ,carbendazim and chlorpyrifos residues and optimize the washing parameters such as solution concentration, temperature and contact time .The effect of the treatments on nutrition value such as the total sugars, total phenolic contents and antioxidant activity in date fruits was also investigated. Scheme of the method showed in figure 1.

## 3. Methodology

### 3.1 Instrumentation

The system of HPLC Agilent 1200 with DAD detector, Column: Agilent Zorbax XDB C18 5um 4.6 x 250mm 5 µm.
UV spectra were recorded on Cary 50 UV-VIS spectrometer using UV Varianse software.
2.2. Chemicals
HPLC grade acetonitrile, and Methanol were obtained from Merck (Germany), pesticides standards were of high purity from Sigma-Aldrich, PestanalTM. Dimethoate (98.0%), carbendazim (98.9%), chlorpyrifos, (98%). Anhydrous sodium acetate, magnesium sulfate was from MERCK, KGaA, EMSURE® and glacial acetic acid were purchased from Fisher Scientific).

DPPH, folin Ciocalteu reagent, Gallic acid, Sodium carbonate were purchased from Fisher. concentrated sulfuric acid 98% was from Merck.

### 3.2 Preparation of Pesticide's Solution

A mixture of pesticide solution contains 20µg/mL of the following pesticides: Dimethoate, chlorpyrifos, and carbendazim was prepared .500 g of Mariami date fruits was washed with deionized water for 2minuts and then immersed in the pesticide's solution for five minutes prior to air drying for 2 hrs.

### 3.3 Preparation of Washing Solutions

Different concentrations of the following washing solutions; tap water, acetic acid and sodium bicarbonate were prepared in deionized water according to the methods described by Satpathy.G et al.,2012, Khaled et al.,2015, Nowowi et al., (2016) and Tianxi et al.,2017.

### 3.4 Washing of Contaminated Samples

The contaminated samples were divided into two groups. group one (A)was kept as controls. Group two (B) were soaked in the washing solutions for intervals of (2,5, and10 min), after it gently rotated by hand for 15-second to simulate the actual washing method practiced in the households. Date samples were dried by air for 2 hr. and divided into two groups. A group (B1) was kept for determination of washing effect on nutrition values while, group (B2) was extracted for pesticide residue analyses.

### 3.5 Extraction of Samples for Pesticide Residue Analysis

#### 3.5.1 Extraction Procedure and Analysis

Date samples (5 g) were weighed in 50 mL centrifuge tubes and then 10 mL of acetonitrile (containing 0.75% glacial acetic acid) was added. After strongly hand shaken for 2 min, 6 g of anhydrous magnesium sulfate and 1.5 g of anhydrous sodium acetate were added. Then the tubes shake again for 30 s and centrifuged at 4000 rpm for 5 min. For clean-up: 1- 2mL of the supernatant was put into another 15 ml centrifuge tube. For each I mL extract, mixture of 50 mg of PSA, 50 mg C18 and 150 mg anhydrous MgSO4 was added. The mixture was vortexed and then centrifuged for 5min at 5000. Finally, 0.5 to 1.0 mL of the supernatant was filtered through 0.2 µm syringe filter and kept in HPLC vail for analysis.
HPLC-DAD was used for pesticide analysis with mobile phase: 95%MeOH+ 5mM Ammonium formate 5% H2O + 5mM Ammonium formate, Injection volume: 20ul. Flow rate: 0.2ml/min.

#### 3.5.2 Recovery

For recovery study, date fruit samples (5 g) were spiked with 0.05,0.10 and 0.15 mg/kg of pesticide's standard solutions in methanol. Then extracted using the modified QuEChERS technique explained earlier. The recovery and precision were evaluated by analyzing the three samples using the following equation:

**Recovery = [concentration in spiked sample – concentration in unspiked sample / concentration in spiked sample] *100**

The recoveries values for the examined ranged from 87 % to 102 %, and RSDs were below 10 %. Limit of detection (LOD) and limit of quantitation (LOQ) were calculated from the signal-to-noise ratios obtained by analyzing unspiked samples. LOD and LOQ were taken to be the concentrations of pesticide resulting in a signal-to noise ratio of 3 and 10, respectively (data not shown).

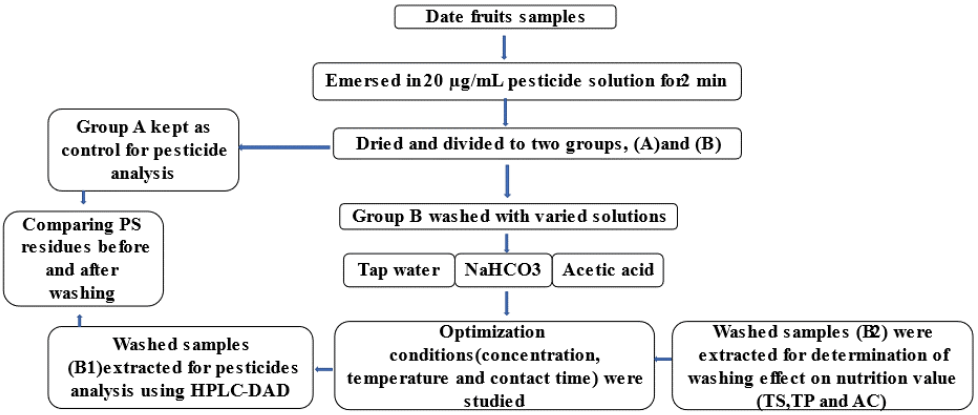


**Figure 1: Scheme of washing process for elimination of pesticide residues from date fruits and determination of the treatment's effect on nutrition value.**

## 4. Determination of washing effect on date fruit's nutrition value

The effect of washing on date fruit's nutrition value was studied throughout the following factors: Total saccharides, total phenolics and the antioxidant activity (scavenging activity of DPPH free radicals).

### 4.1 Determination of Total Sugars and Phenolics Contents

#### 4.1.1 Extraction of Sugars and Phenolics

The extraction procedure was modified slightly from that described by Biglari et al., (2008). Date fruits were sliced into small pieces. One gram of the small pieces was extracted with 10 mL of water at 25°C overnight. The extracts were filtered and refrigerated for further testing.

#### 4.1.2 Determination of Total Sugars

The Sulfuric Acid–UV method explained by Albalasmeh, et al.,2013 was used with some modification as follows. A 1 mL of concentrated sulfuric acid is rapidly mixed with 1 mL of filtered date water extract(100mg/mL) or glucose standard solution in a test tube and vortexed for 30 s. Because the reaction temperature rises quickly within10–15 s after addition of sulfuric acid, the solution was immediately cooled in ice bath for 2 min to return it to room temperature. Finally,2mL of distilled deionized water was added to dilute the residual acid and stope reaction. The UV absorbance at 315 nm was read using UV spectrophotometer. Blank solution was prepared using the same method as above, except that the dates extract was replaced with deionized water. The standard curve was created using glucose solutions in water at concentrations of 0,20,40,60,80,100 mg/mL. The total sugars amount was calculated from the equation of calibration curve and expressed as glucose equivalent mg/g fresh weight, which is a well-known reference compound.

### 4.1.3 Determination of Total Phenolic Contents

Total phenolics were determined using the Folin Ciocalteu reagent, as described by McDonald et al. (2001). Folin Ciocalteu reagent (3mL 1:10 diluted with water) and aqueous sodium carbonate (3mL, 1M) were mixed with diluted date fruit extract (0.1 mL of 1mg/mL) or phenolic standards. To complete the reaction samples were kept in water bath at 45 °C for 15 min. Determination of total phenolic content was performed calorimetrically at 765nm. The standard curve was created using gallic acid solutions in methanol at concentrations of 0,50,100,150,250,300 mg/L. The total phenolic value was estimated using the calibration curve procedure and reported as gallic acid equivalent mg/g fresh weight).

### 4.1.4 Determination of Antioxidants Activity (DPPH inhibition activity)

The DPPH free radical scavenging activity of date fruits was determined using the method described by Oktay et al (2003). 4 mL of 0.1 mM DPPH-methanol solution was added to 200µL methanolic date extract(1mg/mL) and incubated for 60 min at room temperature away from light. Pure methanol was used as blank. Reading the UV absorbance at 715 nm was used to determine the DPPH inhibition activity. Unwashed sample extract was employed as positive controls. Each experiment was triplicated. The inhibition % was calculated using the following equation.

**Inhibition % = {(absorbance of control – absorbance of test sample)/ absorbance of control} *100**

### 4.2 Statistical Analysis

Statistical analysis for each parameter, treatments were carried out in triplicate. The data were expressed as mean ± SD and analyzed using one-way ANOVA. The significance was defined as a probability of 0.05 or less.

## 5. Result and Discussion

### 5.1 The Effects of Wash Treatments on Pesticide Residues

Using HPLC-DAD, the effects of several wash procedures on pesticide residues in date fruits were examined. These procedures include washing with either tap water or three concentration levels of either acetic acid or sodium bicarbonate (1 %, 2 %, and 3 %) in two efferent temperatures ranging( 25°C to 37°C).

Pesticide concentrations in fruits treated with various wash solutions reduced considerably as contact time increased. Additionally, there were substantial variations in the efficacy of the treatments examined and the control (unwashed sample) to remove pesticides throughout a range of contact periods and temperatures. However, changing the contact time during washing resulted in a significant variation in pesticide removal rates.

Data in table 1 shows the result of washing by tap water for 2 and 5min at 25°C and 37°C. After washing for 2 min at 25°C, the removal rates of the examined pesticides were 1.89 %, 5.38% and 18.22 % for dimethoate, carbendazim and chlorpyrifos, respectively. While at 37°C the removal rates 2.42%, 14.42% and 24.48% for the three pesticides, respectively. Removal rates are significantly different within the time intervals and temperature variation ($P<0.05$). Washing for 5

min at 25°C achieved removal rates, of 26.89 ±1.23%,14.38±2.18 %and 62.22 ± 2.03% for dimethoate, carbendazim and chlorpyrifos, respectively and 44.42± 2.78% ,31.54± 0.32% and 79.17 ± 2.53% removal rates respectively when the temperature was 37°C, compared with the control. The significant differences (P>0.05) between the removal rates at 25°C and 37°C indicated that temperature has a positive effect on pesticides' removal rates by soaking in water. It should be noted that the decrease of pesticides by water washing in this study was more than that reported by Abou-Arab (1999) and Cengiz et al., (2006), who reported decrease rates of 9–23% of pesticide residues by washing fruits with tap water. Additionally, our findings differed from those of Karol et al, (2000) which revealed that soaking fruits and vegetables in tap water for 15–30 seconds significantly reduced pesticide residual levels, including malathion, iprodione, and others. But it is close to the removal rates reported by Liang et al.,2012 (53% and 62.9 % for chlorpyrifos depending on soaking time, and for dimethoate,15.2% ,21.7% and 32.6% at 5,10 and 20min time intervals, respectively). The results also corroborate Osman et al(2014) .'s observation that washing date fruits in flowing tap water removed 37.08 %, 42.46 %, 51.77 %, and 51.93 % of chlorpyrifos residues in 5,15,30, and 60 minutes, respectively. Alternatively, Lee et al., 1991 reported that washing rice grains in water eliminated around 60% of chlorpyrifos residues. Xia E et al., (2016) reported removal rate for carbendazim of >70% from mushroom by rinsing under running water for 5 min and removing rate 53.64 % by Soaking in tap water for ten min.

**Table 1: Removal (%) of pesticides in dates by water for 2 and 5 min at 25C and 37C**

| Tap water | 2min | | 5min | |
|---|---|---|---|---|
| | 25°C | 37°C | 25°C | 37°C |
| Pesticide | Removal % | Removal % | Removal % | Removal % |
| Dimethoate | 10.89 ± 1.23* | 12.42 ± 0.78* | 26.89 ± 1.23* | 44.42 ± 2.78* |
| Carbendazim | 5.38 ± 0.4 * | 14.14 ± 0.52* | 14.38 ± 2.18* | 31.54 ± 0.32* |
| Chlorpyrifos | 18.22 ± 2.03* | 24 .48 ± 2.84* | 62.22 ± 2.03* | 79.17 ± 2.53* |

*Means are significantly different values.

From table 2, sodium bicarbonate 1% (w/v) at 25 °C and three-time intervals of, 2, 5, and 10 min achieved removal rates of dimethoate of 94.89 ± 0.73%, 96.35 ± 0.50 % and 97.88± 0.74 % respectively. At 37°C, the removal % was increased to 95.90± 0.59 %, 98.09± 0.88% and 98.31± 0.34% respectively according to the contact time. The washing time have positive effect on the removal rates. Washing of samples by NaHCO3 2% solution at 25°C for 2,5, and 10 min caused removal rates of dimethoate of 96.189±0.28%, 97.12± 0.51% and 98.13 ± 0.055% respectively while at 37°C, the removal rates by were slightly increased by temperature to become: 98.04±1.16, 98.46± 0.95% and 98.37±1.25%. NaHCO3 3% at 25°C caused removal of dimethoate by rates of 98.96±0.012%, 98.38 ± 0.04% and 98.79 ± 0.23 % at 2,5, and 10 min respectively while at 37°C, the removal rates of dimethoate were almost stable and seems not effected clearly by increasing of soaking time and temperature as the following: 98.67±0.52%, 98.34±0.45% 98.44±0.42%.

Removal rates of carbendazim by 1% NaHCO3 at 25°C were 13.5±5.6, 15.9±0.06 and 21±6.41% at for time intervals of 2,5 and 10 min respectively and 16.5±3.88%, 18.5±1.79 % and 24.9 ±10.3% at 37°C for the same time intervals, respectively.

In samples washed by NaHCO3 2% solution, Carbendazim removal rates at 25°C, were 17.2±1.51%, 21.8±1.07% and 24.5±10.50%, while at 37C the removal rates of carbendazim remained almost the same as following; 18.6±5.78%, 23.6±1.39% and 23.8±12.18 %. While washing by NaHCO3 3% at 25°C carbendazim removing rates were positively affected by soaking time as the following: 21.5±10.55%, 23.6±8.12% and 25.3±2.85 % respectively. At 37°C, carbendazim showed removal rates of 23.2±10.64%, 25.4±7.77 % and 27.5 ± 8.06 % which indicate the positive effect of temperature and soaking time on removal rates of carbendazim.

Chlorpyrifos removal % by NaHCO3 (1%) at 25 °C was 73.11±0.95%b,82.21±8.40 %and 87.48±5.1% for 2,5, and 10 min respectively, while at 37°C the removal rates were 74.87±1.11%, 83.32 ± 2.9% and 88.43 ± 3.69% respectively. NaHCO3 (2%) solution caused Chlorpyrifos removal rates of 83.70±4.45%, 86.02±3.9% and 88.26±3.44% at 2,5 and 10 min respectively at 25°C. At 37°C the removal rates were improved within temperature and washing time to become 85.69±3.64%, 88.78±0.66% and 89.95±0.78% respectively. Washing by NaHCO3 3% solution, Chlorpyrifos was approximately 100 % removed at all time intervals and not detected during sample analysis.

However, Increasing the concentration of $NaHCO_3$ solution from 1%,2% and 3%, gave a positive effect on removal rates of carbendazim, chlorpyrifos and dimethoate. Dimethoate removing rates was the highest among the group at all experimental conditions.

However, Liang et al.,2012 stated that between detergent solutions, both carbonate and bicarbonate at concentration 2% and 5%(w/v), had the greatest loss in organophosphorus pesticides and reported removal rates for chlorpyrifos as 75,3%, 77.8 %and 81.5% at 2,10 and 20 min respectively. In addition, the study concluded that the soaking with detergent solutions has also been shown to be more efficient than tap water.

Acetic acid effect on pesticides removal at different concentrations, temperature and time intervals was studied. Data in table.3 showed that acetic acid (AA)1% at 25°C exhibited high removal rates of dimethoate as 73.49 ± 1.53%, 81.89 ± 0.98 % and 85.15 ± 3.49% for the time intervals 2,5, and 10 min respectively while at 37°C, the removal rats were increased to become 86.74±5.87, 86.29±5.67, and 88.98 ± 1.23 % respectively for the same time intervals. At concentration 2%, acetic acid showed higher removal rates at both 25 and 37 °C. For dimethoate, the removal rates at 25°C were 83.85±3.81, 86.21±1.32 and 91.33±1.05 at 2,5,10 min respectively and 92.23±1.95, 91.01±0.73 and 93.57±1.47% at 37°C at the same time intervals, respectively. Washing with 3% acetic acid at 25°C, the removal rates of dimethoate were 94.94±4.52, 95.14±5.68 and 96.42±2.12 at 2,5- and 10-min time intervals, respectively. At 37°C, removal rates were 96.45±1.8, 96.28±2.11 and 96.74±2.42 at 2,5, and 10 min, respectively.

In case of carbendazim, acetic acid 1% showed low removal rates of 8.15±0.33, 10.95±1.35 and 11.37±1.36 % at 25 °C and 11.22 ± 0.46,12.3 ± 2.35 and 15.28± 0.56% at 37 °C. However, both temperature and soaking time are facilitating the removal rates of pesticides. Acetic acid 2%, at 25°C achieved carbendazim removal rates as the following: 17.83±2.59, 20.15±3.50 and 20.38±5.43 at time intervals 2,5, and 10 min respectively while at 37°C the removal rates were 20.50±7.91, 22.53±7.42 and 20.11±3.78 at time intervals 2,5, and 10 min respectively. With 3% acetic acid at 25°C, carbendazim removal rates were 20.3±7.45, 24.25±4.30 and 20.38±5.43 at

2,5, and 10 min respectively, while at 37°C it removed by rates of 23.01±9.7, 25.3±6.10 and 31.9±1.27 respectively. The removal rates of chlorpyrifos were 88.72±1.73, 89.63±0.37 and 91.48±3.31 when washing temperature was 25°C. While at 37°C, the removal rates were 92.53±3.09, 94.45±3.35 and 97.25±0.45 at same time intervals, respectively.

Washing by acetic acid 1% at 25 °C, chlorpyrifos was removed by rates of 75.88 ±0.47, 78.03±0.87 and 80.93 ± 1.23 and time intervals of 2,5,10 min, respectively. while at 37°C, chlorpyrifos removed by rates of 85.63 ± 4.74%, 79.85 ± 0.44 % and 82.68 ± 2.17% respectively under the same experimental conditions of concentration and soaking time. The removal rates of Chlorpyrifos by acetic acid 2% at 25C were 83.09 ± 3.16, 86.82 ± 5.48 and 87.87± 1.75 %. However, at 37°C chlorpyrifos was removed by 86.25 ± 0.45, 87.13 ± 0.63 and 88.54 ± 0.98, respectively. With 3% acetic acid at 25°C, the removal rates of Chlorpyrifos removal rates were 88.72±1.73, 89.63±0.37 and 91.48±3.31 when washing temperature was 25C. While at 37°C, the removal rates were 92.53±3.09, 94.45±3.35 and 97.25±0.45 at same time intervals, respectively. From the result it clear that all examined factors such as acetic acid concentration, temperature and contact time has a positive effect on pesticides removal. Through the obtained results, all the parameters examined (solution concentration, soaking temperature and, time intervals exerted a positive effect on removal rates of pesticides. However, profenofos was completely eliminated from eggplant when it immersed in a 1% soap solution or a 2% acetic acid solution (Radwan et al,.2005). Similarly, the study conducted by Kin, C. M., & Huat, T. G. 2010 reported that acetic acid removed 44–70% of residues from samples, followed by sodium carbonate ( 30–50%) removal rate and salt (23–40%) removal rate of residues. furthermore, Osman et al., 2014 stated rinsing date fruits by 1% acetic acid solution removed 81.12, 88.91, 91.93 and 98.42% of chlorpyrifos residue while rinsing at concentrations of 2% improved the efficiency of pesticide removal at 87.23%, 93.36%, 99.17% and 99.5 % respectively at 5, 15, 30 and 60 min.

On the other hand, Pugliese et al. 2004 reports that nectarines spiked with pesticides namely, chlorpyrifos, iprodione, fenarimol, malathion, myclobutanil, methidathion, pirimicarb and parathion treated with solutions of sodium hypochlorite, Citric acid, $H_2O_2$, $KMnO_4$, urea and sodium metabisulfite provided results like those obtained with tap water and no significant differences were found in removal rates comparing with tap water. The result of the present study agrees with those obtained by Osman et al 2014. Osman et al. (2014) discovered that chlorpyrifos is more easily removed from date fruit following treatment with the investigated chemical solutions as compared to running water. Their findings indicated that the removal of chlorpyrifos increased in the order acetic acid > citric acid > hydrogen peroxide (H2O2)> potassium permanganate (KMnO4)> running water, and that the percentage of pesticide residue removed from date fruit was also dependent on the solution concentration and dipping time. Washing with water or soaking in salt solutions, as well as the use of specific chemicals, such as chlorine, chlorine oxide, ozone, hydrogen peroxide, acetic acid, hydroxyl peracetic acid, iprodione, and specific detergents, have been shown to be quite effective at decreasing pesticide levels (Bajwa and Sandhu 2014).In the present study dimethoate removal rates is higher than that of chlorpyrifos that may refers to its solubility in water >1000µg/L at 20°C while solubility of chlorpyrifos is <10 µg/L which considered very poor. but the solubility rule is not working with carbendazim which have solubility 29 mg /L and showed the lowest removing rates with all examined chemicals (mostly <40%). The low removal rates of carbendazim may attributed to being penetrating very fast to deep date's tissues and could not be removed within the applied contact time. According to

Angioni et al.,2004, there is no correlation between pesticides' water solubility and their rate of degradation following washing. Burchat et al., 1998 highlighted that the behavior of pesticides during washing vary not only by pesticide type, but also by crop type. This was proved by the quantity of pesticide residue removed from two different vegetables after they were washed (tomatoes and carrots). Moreover, the removal rates of pesticides from carrots were higher than that from tomatoes. This might be because pesticides are partially partitioned in the waxy layers of tomato skin. The results showed that the removal of dimethoate and chlorpyrifos from date fruits is dependent on the concentrations of tested chemical solutions, temperature, and contact times, which is similar to the findings of Abou-Arab (1999), who found that the loss of HCB, lindane, p,p'-DDT, dimethoate, profenofos, and pirimiphos-methyl is dependent on acetic acid and NaCl concentrations. Furthermore, the amount of pesticide eliminated by washings is related to its solubility in water and the octanol–water partition coefficient (Pugliese et al., 2004) with exception of carbendazim behavior in this work which showed lower removal rate and less affect by changing the optimization factors (concentration, contact time and temperature) which may attributed to the ability of carbendazim to penetrate through date fruit tissues.

**Table 2: Removal (%) of pesticides in dates by sodium bicarbonate NaHCO3**

| **NaHCO3** | **Removal rates %** | | |
|---|---|---|---|
| | **Dimethoate** | **Carbendazim** | **Chlorpyrifos** |
| NaHCO3.1%.2min.25C | 94.89 ± 0.73* | 13.5±5.6 x | 73.11±0.95* A |
| NaHCO3.1%.5min.25C | 96.35± 0.50 | 15.9±0.06 | 82.21±8.40 |
| NaHCO3.1%.10min.25C | 97.88± 0.74 | 21±6.41 | 87.48±5.1* |
| NaHCO3.1%.2min.37C | 95.90± 0.59 | 16.5±3.88 | 74.87±1.11 |
| NaHCO3.1%.5min.37C | 98.09± 0.88* | 18.5±1.79 | 83.32±2.9 |
| NaHCO3.1%.10min.37C | 98.31± 0.34* | 24.9±10.3 | 88.43±3.69 B |
| NaHCO3.2%.2min.25C | 96.189±0.28 | 17.2±1.51 | 83.70±4.45 |
| NaHCO3.2%.5min.25C | 97.12±0.51 | 21.8±1.07 | 86.02±3.9 |
| NaHCO3.2%.10min.25C | 98.13±0.05* | 24.5±10.50 | 88.26±3.44 |
| NaHCO3.2%.2min.37C | 98.04±0.16* | 18.6±5.78 | 85.69±3.64 |
| NaHCO3.2%.5min.37C | 97.46±0.95* | 23.6±1.39 | 88.78±0.66 |
| NaHCO3.2%.10min.37C | 98.37±1.25* | 23.8±12.18 | 89.95±0.78 |
| NaHCO3.3%.2min.25C | 98.96±0.012* | 21.5±10.55 | 100 |
| NaHCO3.3%.5min.25C | 98.38±0.04* | 23.6±8.12 | 100 |
| NaHCO3.3%.10min.25C | 98.79±0.23* | 25.3±2.85 | 100 |
| NaHCO3.3%.2min.37C | 98.67±0.52* | 23.2±10.64 | 100 |
| NaHCO3.3%.5min.37C | 98.34±0.45* | 25.4±7.77 | 100 |
| NaHCO3.3%.10min.37C | 98.44±0.42* | 27.5± 8.06 | 100 |

**Table 3 Removal (%) of pesticides in dates by acetic acid (AA)**

| Acetic acid | Removal rates % | | |
|---|---|---|---|
| | **Dimethoate** | **carbendazim** | **Chlorpyrifos** |
| AA1%,2min,25°C | 73.49±1.53 | 8.15±0.33 | 75.88 ±0.47 |
| AA1%,5min,25°C | 81.89±0.98 | 10.95±1.35 | 78.03±0.87 |
| AA1%,10min,25°C | 85.15±3.49 | 11.37±1.36 | 80.931.22 |
| A1%,2min,37°C | 86.74±5.87 | 11.22±0.46 | 85.63±4.74 |
| AA1%,5min,37°C | 86.29±5.67 | 12.3±2.35 | 79.85±0.44 |
| AA1%,10min,37°C | 88.98±1.23 | 15.28±0.56 | 82.68±2.17 |
| AA2%,2min,25°C | 83.85±3.81 | 17.83±2.59 | 83.09±3.16 |
| AA2%,5min,25°C | 86.21±1.32 | 20.15±3.50 | 86.82±5.48 |
| AA2%,10min,25°C | 91.33±1.05 | 20.38±5.43 | 87.87±1.75 |
| AA2%,2min,37°C | 92.23±1.95 | 20.50±7.91 | 86.25±0.45 |
| AA2%,5min,37°C | 91.01±0.73 | 22.53±7.42 | 87.13±0.63 |
| AA2%,10min,37°C | 93.57±1.47 | 23.11±3.78 | 88.54±0.98 |
| AA3%,2min,25°C | 94.94±4.52 | 20.3±7.45 | 88.72±1.73 |
| AA3%,5min,25°C | 95.14±5.68 | 24.25±4.30 | 89.63±0.37 |
| AA3%,10min,25°C | 96.42±2.12 | 25.96±7.57 | 91.48±3.31 |
| AA3%,2min,37°C | 96.45±1.8 | 23.01±9.7 | 92.53±3.09 |
| AA3%,5min,37°C | 96.28±2.11 | 25.3±6.10 | 94.45±3.35 |
| AA3%,10min,37°C | 96.74±2.42 | 31.9±1.27 | 97.25±0.45 |

## 5.2 Effect of Different Wash Solutions on Nutrition Value of Date Fruits

From table.4 sample washed with water at 25 for 2 min give TS of 700.97 mg/g caused reduction of total sugars by 10.8 %, while sample washed at 37°C for the same time interval showed TS content of 634.25mg/g with 19.2% reducing rate compared with control. The result indicates that the temperature has significant effect on TS reducing with water soaking ($P<0.05$) when solution concentration and washing time were fixed Data table.5.8

Sample washed by water at 25°C for 5 min give TS of 667.4 mg/g caused reduction of total sugars by 21.79 %, while sample washed at 37°C for the same time interval showed TS content of 526.5 mg/g with 32.12% reducing rate compared with control. The result indicates that the temperature has significant effect on TS reducing with water soaking. Result showed the time changing from 2 to 5 min has a significant effect on TS reduction % ($P<0.05$) when the temperature was fixed.

**Table 4: Total sugars contents and reduction % of samples washed by water mg/g glucose equivalent compared to control sample.**

| Sample | TS mg/g | Rd% |
|---|---|---|
| Unwashed(control) | 775.7 ± 29.14 | - |
| Tap water.2min 25°C | 700.97 | 10.8 |
| Tap water.2min 37°C | 634.25 | 19.32 |
| Tap water.5min 25°C | 676.4 ± 11.92 | 12.80 |
| Tap water.5min 37°C | 526.47 ± 16.13 | 32.13 |

Washing with Sodium bicarbonate result table.5, exerted very little effect on TS. The effect slightly increased with, temperature, contact time, and concentration from 0. 5% for concentration of 1%, 2min at 25°C to 7.5% for concentration of 3% ,10min at 37°C. Washing with acetic acid resulted in very little effect on TS also. Decreasing in TS content slightly increased with, temperature, contact time, and concentration from 0.75 % for concentration of 1%, 2min at 25°C to 4.92 % for concentration of 3% ,10 min at 37°C. The effect increased with, contact time and concentration from 4 % for concentration of 1%, 2min at 25°C to 12.3% for concentration of 3% ,10 min at 25°C. By increasing the contact period, the sugar loss from date fruits was increased. The findings are consistent with those of Osman et al. (2014), who discovered that acetic acid, citric acid, hydrogen peroxide, and potassium permanganate all had a detrimental effect on total sugars. According to the study, soluble sugars are easily dissolved in washing solutions and are continually lost from dates. Time and temperature are important considerations in the washing process.

**Table 5: Total sugars contents and reduction % of samples washed by different concentrations of sodium bicarbonate and acetic acid at 25°C and 37°C and time intervals of 2,5 and 10 minutes. values are mean ± DS.**

| Solution | NaHCO3 | | Acetic acid | |
|---|---|---|---|---|
| | TS | Rd % | TS | Rd % |
| 1%2min25°C | 772.59 | 0.39 | 769.8 | 0.76 |
| 1%5min25°C | 768.5 | 0.92 | 763.56 | 1.56 |
| 1%10min25°C | 755.5 | 2.6 | 754.36 | 2.75 |
| 1%2min37°C | 769.77 | 0.76 | 762.66 | 1.68 |
| 1%5min37°C | 762.04 | 1.76 | 757.41 | 2.36 |
| 1%10min37°C | 751.80 | 3.08 | 749.53 | 3.38 |
| 2%2min25°C | 770.14 | 0.71 | 765.81 | 1.28 |
| 2%5min25°C | 765.79 | 1.28 | 765.47 | 1.32 |
| 2%10min25C | 753.45 | 2.88 | 759.88 | 2.05 |
| 2%2min37°C | 767.11 | 1.10 | 760.88 | 1.91 |
| 2%5min37°C | 763.70 | 1.55 | 759.733 | 2.06 |
| 2%10min37°C | 748.9 | 3.45 | 753.69 | 2.84 |
| 3%2min25°C | 758.79 | 2.18 | 756.60 | 2.46 |
| 3%5min25°C | 731.1 | 5.75 | 754.58 | 2.72 |
| 3%10min25C | 738.04 | 4.85 | 742.22 | 4.315 |
| 3%2min37°C | 746.95 | 3.71 | 750.920 | 3.19 |
| 3%5min37°C | 723.73 | 6.69 | 745.81 | 3.85 |
| 3%10min37°C | 717.53 | 7.5 | 737.47 | 4.93 |

As shown in table 6, Samples washed by tap water for 2min at 25°C, showed TP contents of 1.57mg (GAEQ)/g weight of fresh sample compared to unwashed sample which showed TP content of 1.64 mg (GAEQ)/g with reduction% of 4.02% in TP contents. the sample washed at 37°C for the same time interval(2min), TP content was found 1.525 mg/g with reduction rate of 7%.the result indicated the significant effect of the temperature on TP content decreasing during washing. Samples washed by water for 5min at 25°C, gave TP content of 1.48 mg/g with reduction rate of 9.42% which is significantly deferent ($P<0.05$) from that of sample washed for 2min at

25°C.While sample washed at 37C for 5 min gave TP content of 1.43 mg/g with reduction rate of 12.86% compared to control sample. Changing washing temperature causing very significant decrease in TP contents at same washing time (P<0.001). Both time and temperature have significant effect on TP content during water washing.

**Table 6: effect of tap water at 25°C and 37 °C on total phenolic contents 2 and 5 min.**

| Sample | TP. contents | Reduction % |
|---|---|---|
| Control sample | 1.64 ± 0.054 A | |
| Tap water 2min 25°C | 1.574 ± 0.15 | 4.015 |
| Tap water 2min 37°C | 1.525 ± 0.087 | 7.002 |
| Tap water 5min 25°C | 1.48 ± 0.06 B | 9.42% |
| Tap water5min 37°C | 1.43 ± 0.028 B | 12.86% |

**Table 7: Total phenolic contents and total phenolic reduction % in date's samples washed by NaHCO3and acetic acid solutions at 25°C and 37 °C for 2, 5 and 10 min compared to unwashed sample.**

| Washing solution | NaHCO3 | | AA | |
|---|---|---|---|---|
| | TP | Rd% | TP | Rd% |
| 1%2min25°C | 2.19 | 0.32 | 2.23 | 1.4 |
| 1%5min25°C | 2.2 | 0.46 | 2.12 | 3.53 |
| 1%10min25°C | 2.12 | 0.360 | 2.09 | 4.95 |
| 1%2min37°C | 2.18 | 0.74 | 2.21 | 4.51 |
| 1%5min37°C | 2.17 | 1.54 | 2.11 | 4.12 |
| 1%10min37°C | 2.12 | 3.69 | 2.09 | 5.07 |
| 2%2min25°C | 2.18 | 0.86 | 2.19 | 0.62 |
| 2%5min25°C | 2.18 | 1.09 | 2.105 | 4.32 |
| 2%10min25°C | 2.12 | 3.69 | 2.04 | 7.06 |
| 2%2min37°C | 2.18 | 0.99 | 2.06 | 6.35 |
| 2%5min37°C | 2.15 | 2.03 | 2.043 | 7.11 |
| 2%10min37°C | 2.11 | 4.15 | 2.02 | 8.34 |
| 3%2min25°C | 2.17 | 1.07 | 2.055 | 6.57 |
| 3%5min25°C | 2.11 | 3.91 | 2.02 | 8.01 |
| 3%10min25°C | 1.78 | 19.25 | 1.96 | 10.85 |
| 3%2min37°C | 1.73 | 21.18 | 2.03 | 7.57 |
| 3%5min37°C | 1.65 | 24.98 | 2.02 | 8.23 |
| 3%10min37°C | 1.62 | 26.24 | 1.88 | 14.17 |

Samples washed with NaHCO3(Table 7) showed gradual decreasing in TP contents. The effect of washing increased with contact time, temperature, and concentration of NaHCO3 solution.TP contents decreased from 0.32 % by concentration 1% for 2min and 25°C to reach the maximum decreasing effect of 26.24 % at concentration of 3% for 10min and 37°C. At concentration of 3%, washing has drastic effect on TP specially at 37°C which caused 26.24% reduction rate while concertation of 1% and 2% mostly did not exceed reduction rate of 4%.

From data table 7, the effect of acetic acid solutions on TP decreasing was ranged from 1.4 % at concentration 1%, for 2 min at 25°C to 14.17% at concentration of 3% for 5min and 37°C. Changing in Temperature, contact time and solution concentration resulted in a significant (P>0.05) decreasing in TP contents (increasing reduction rate of TP).

The effect of washing on (antioxidant activity) DPPH inhibition activity was tested. The results table 8 and table.9 showed gradual decreasing in antioxidant activity of tested samples comparing with the control (unwashed sample. The rate of DPPH inhibition reduction was affected according to the washing solution, concentration, temperature, and contact time.

As shown in table.8 and table 9, all extracts of washed samples demonstrated inhibitory activity against the DPPH radical compared to that of unwashed sample (100 mg/ml). Sample washed by water for 5 min at 25 °C showed DPPH inhibition activity of 75.8% with 11.5% reduction rate compared with 85% inhibition % exerted by control sample. While sample washed at 37°C showed DPPH inhibition of 68.6 % with (19.9% reduction) compared with unwashed sample activity.

**Table 8: Inhibition and inhibition reduction% of DPPH of samples washed by tap water and controls (unwashed sample) at 25 and 37C and time intervals of 2 and 5 minutes. values are mean ± D. values with different litters are significantly different (P < 0.05).**

| Sample | Inhibition % | Rd% |
|---|---|---|
| Tap water 2min at 25C | 83.34 a | 2.7±0.54 |
| Tap water2min at 37C | 71.87 b | 16.09 ± 3.1 |
| Tap water 5min at 25°C | 75.80 ± 1.95 c | 11.5 |
| Tap water 5min at 37°C | 68.60 ± 3.83 d | 19.9 |

Samples washed with 1% NaHCO3 solution at 25°C showed the lowest effect on DPPH inhibition activity which started with 72.64% with reduction rate of 2.3 % ,72.2% with reduction rate 3.15%, and 71.72% with reduction rate of (3.56%) for 2,5 and10 min time intervals, respectively. There was no significant difference between inhibition activities measured at the three-time intervals at 25°C and the inhibition activity of the control (P>0.05). While at 37°C the DPPH inhibition activity of washed samples was significantly decreased (P<0.05) to become 65.85 with reduction rate of 11.44%,64.73 with inhibition rate of 12.95% and 66.36% with reduction rate of 10.76 % for the same time intervals, respectively. But there was no significant difference in inhibition activities among samples washed for 2-, 5- and 10-min time intervals at 37°C (P>0.05).

Washing with 2% NaHCO3 solution at 25°C showed significant decreasing in DPPH inhibition activity compared with control (P<0.05) as following; 65.25% with reduction rate of 12.26, 64.97% with reduction rate of 12.62% and 62.22% with reduction rate of 16.33% for 2,5and 10 min time intervals, respectively. The inhibition activity within 2- and 10-min intervals was significantly different (P<0.05). While at 37 C the DPPH inhibition activity was 60.38% with reduction rate of 18.79%,63.94% with reduction rate of 14.01%, and 65.50% with reduction rate of 11.91% for the same time intervals, respectively. The inhibition activity at 2 min time interval and 37°C was significantly different from that at 25°C and the same time interval, indicating that temperature increasing has a significant effect on decreasing DPPH inhibition activity.

Samples washed with 3% NaHCO3 solution at 25°C showed more significant decreasing in DPPH inhibition activity compared with control($P<0.05$) as following; 63.46%with reduction rate of 14.28%, 62.26% with reduction rate of 16.26 % % and 62.68% with reduction rate of 15.71% for 2,5- and 10-min time intervals, respectively. The was difference in the effect of washing for 2 and 10 min at 25°C was significant. At 37°C the inhibition activity was 59.89% with reduction rate of 19.46%, 58.31% with reduction rate of 21.58%, and 57.21% with reduction rate of 23.06% for the same time intervals, respectively. However, at 37°C the decreasing in inhibition activity after 2,5 and 10 min was very significant ($P<0.001$). The difference in decreasing of DPPH inhibition activity at 25C and 37C at 3% concentration and 2,5- and 10-time intervals was also significant ($P<0.05$). all studied parameters i.e., solution concentration, temperature, and contact time exerted significant effect on DPPH inhibition activity in most cases.

Samples washed with 1% acetic acid (AA) solution at 25°C showed the lowest reduction effect on DPPH inhibition activity start with 74.05 with reduction rate of 0.411%, 72.35 with reduction rate of 2.71%and 68.46% with reduction rate of 7.9% for 2.5-, and 10-min time intervals, respectively. While at 37°C the DPPH inhibition activity was 72.18% with reduction rate of 2.93% ,72.22 % with reduction rate 2.88% and 66.90% with reduction rate of 10.03% compared to control sample inhibition activity for the same time intervals, respectively. There was no significant deference between the DPPH inhibition activities of washed samples ($P>0.05$) when the AA concentration of 1% under all optimization conditions.

Samples washed with 2 % acetic acid (AA) solution at 25°C showed lower DPPH inhibition activity and higher reduction rates as following; 67.42% with reduction rate of 9.3%, 68.64% with reduction rate of 7.7 % and 65.98% with reduction rate of 11.26% for 2.5- and 10-min time intervals, respectively. Inhibition activity was significantly reduced in the sample washed for 10 min at 25°C compared to control. While at 37°C the DPPH inhibition activity was 66.86% with reduction rate of 10.08 % ,63.91 % with reduction rate of 14.05 and 50.71 % with reduction rate of 31.8 % compared to control sample inhibition activity for the same time intervals, respectively. Samples washed with 2% AA solution for 5and 10 min, significantly reduced the inhibition activity compared control and compared samples washed with 1% AA solution under all optimization condition which indicated the effect of concentration of AA is more dominant than the effect of temperature and washing time.

Samples washed with AA 3% at 25C showed DPPH inhibition activity of 58.58% with reduction rate of 21.22, 57.77% with reduction rate of 22.31% and 52.73% with reduction rate of 29.09% for 2,5- and 10-min time intervals, respectively. While at 37°C the DPPH inhibition activity was 51.99% with reduction rate of 30.07% and 46.07% with reduction rate of 38.04% and 42.8% with reduction rate of 42.44% for the same time intervals, respectively. No significant difference in DPPH inhibition activities of samples washed with 3% AA solution under the studied optimization conditions at 25°C but, the difference became significant ($P<0.05$) when washing was performed at 37°C for 2,5 and10 min time intervals.

However, the effect of washing solutions on TS for 5 min seemed to be in descending order as follows: H2O>AA>NaHCO3.The effect of washing solutions on TP is illustrated in tables, 6 and 7 indicated that all treatments resulted in gradual decreasing in TP contents. Increasing in contact time, temperature or concentration of washing solution led to more decreasing in TP contents and

revealed significant differences ($p < 0.05$) in phenolic content between samples washed for different time intervals different temperatures and different solution concentration but the effect of temperature and solution concentration is greater than the effect of contact time. In general, the washing solutions effect on TP contents was in descending order as follows: water > AA>NaHCO3.

However, the effect of washing on antioxidant activity AC (DPPH inhibition activity) was following a similar trend as TP, this may because AC is mostly referring to total phenolic contents. It is commonly known that antioxidant activity is mainly refer to the presence of constituents with high free radical scavenging activity such as phenolics and flavonoids (Biglari et al.,2008, Osman et al.,2014). Strong correlation between TP contents and antioxidant activity AC in date fruits was established and reported by many researchers (Allaith 2008, Biglari et al 2008). Similar results were reported in the study conducted by Nasrin et al.,2015 on vegetables cleaned solely with water or with different combinations of water and detergent and calcium hypochlorite solution. The initial analyses were performed immediately after washing on day one, and further analyses were performed after 3 and 5 days of storage at 4°C. The effect of washing on antioxidant activity varied depending on the technique.. In comparison with other washing treatments, washing in order with water, detergent, and calcium hypochlorite was significantly ($P < 0.05$) decreased antioxidant activity.

**Table 9: DPPH inhibition activity and reduction % compared with unwashed sample inhibition activity (74.22 %) of samples washed by NaHCO3 and acetic acid.**

| | NaHCO3 | | AA | |
|---|---|---|---|---|
| **Washing solution** | **Inhibition%** | **Rd%** | **Inhibition%** | **Rd %** |
| 1%2min25°C | 72.64 | 2.3 | 74.05 | 0.411 |
| 1%5min25°C | 72.02 | 3.15 | 72.35 | 2.71 |
| 1%10min25°C | 71.72 | 3.56 | 68.46 | 7.9 |
| 1%2min37°C | 65.85 | 11.44 | 72.18 | 2.93 |
| 1%5min37°C | 64.73 | 12.95 | 72.22 | 2.88 |
| 1%10min37°C | 66.36 | 10.76 | 66.90 | 10.03 |
| 2%2min25°C | 65.25 | 12.26 | 67.42 | 9.3 |
| 2%5min25°C | 64.97 | 12.62 | 68.64 | 7.70 |
| 2%10min25°C | 62.22 | 16.33 | 65.98 | 11.26 |
| 2%2min37°C | 60.38 | 18.79 | 66.86 | 10.08 |
| 2%5min37°C | 63.94 | 14.01 | 63.91 | 14.05 |
| 2%10min37°C | 65.50 | 11.91 | 50.71 | 31.80 |
| 3%2min25°C | 63.46 | 14.28 | 58.58 | 21.22 |
| 3%5min25°C | 62.26 | 16.26 | 57.77 | 22.31 |
| 3%10min25°C | 62.68 | 15.71 | 52.73 | 29.09 |
| 3%2min37°C | 59.89 | 19.46 | 51.99 | 30.07 |
| 3%5min37°C | 58.31 | 21.58 | 46.07 | 38.04 |
| 3%10min37°C | 57.21 | 23.06 | 42.8 | 42.44 |

## 6. Conclusion

Washing using tap water and other nontoxic chemical solutions is critical for reducing pesticide residues in fruits. Since the concentration of the washing solution, the duration of the treatment, and the temperature all have an effect on the efficacy of pesticide removal, the removal rate may be increased by optimizing these parameters. The administered wash treatments substantially eliminated the identified pesticides from date fruits at all time intervals compared to unwashed fruits (control). The washing temperature has significant positive effect on pesticide removal especially with dimethoate and chlorpyrifos in case of water, acetic acid, and sodium bicarbonate. Carbendazim removal rates were the lowest through all the treatments which may related its ability to faster penetrating the date tissues. Almost complete removal of dimethoate and chlorpyrifos occurred in sodium bicarbonate, the removal rates of acetic acid washed fruits were ranging from 73.49- 96.74 and from 73.11-97.25 for dimethoate and chlorpyrifos, respectively. Although washing by tap water dramatically decreased pesticide residues on date fruits as compared to a control sample, it is still regarded less efficient than other washing procedures. Pesticides' poor water solubility and high octanol water partition coefficient may account for their low removal rates. Furthermore, the results of the current study demonstrated that the decrease % was increasing gradually because of increasing the concentrations of washing solution, contact duration, and temperature. All treatments had a detrimental effect on the nutritional content of the date fruits, as concentration, washing time and temperature increased. However, the increasing in temperature showed the highest effect on nutrition value. To reduce the effect of washing on date fruits nutrition value, the treatment should be carried out for about 5 min at 25°C or less and the washing solution concentration should be 1 or 2 %.

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