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Preparation OF Active Carbon from Date and Olive Stones and Its Contribution on Virus Removal from water

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Abstract

Use of date and olive stones as a raw material available in large quantity in Egypt for producing a low cost activated carbon and characterization in this work. The preparation method entails the impregnation of the dried and crushed stones with activated agent $ZnCl_2$ followed by carbonization at high temperature. The quality of the activated carbon prepared was identified by Physical and chemical characterization. The both prepared activated carbon has very large surface area as well as highly developed micro porosity, with good capacity for iodine and methylene blue removal. The objective of this research was to investigate the potential of the two prepared and commercial available activated carbon as sorbent for removing viruses from water and to delineate the sorption mechanism. Powdered activated carbon successfully removed it under the same condition than granular form with priority for our prepared from stones. Three factors contributed to virus removal: a smaller electrophoretic repulsive force between the virus and the activated carbon particles, a small proportion of pores 1.6 and 3.7 nm in diameter, and a greater hydrophobicity of the virus surface.

Keywords: Activated carbon, Date and Olive Stones, Virus Removal, Repulsion force, hydrophobicity.

1. Introduction

Water is an important and essential component of this universe and plays a vital role in the proper functioning of the Earth's ecosystems. In spite of this, safe drinking water is not available in some parts of the world. The quality of water resources is deteriorating exponentially because of their contamination [1]. Pure water does not occur in nature. It contains impurities which causes water pollution. According to WHO 80% disease are water borne. Contaminated water causes harmful effect on human health and environment [2]. Both point and non-point sources are polluting our water resources as a result of tremendous population growth, modern industrialization, civilization, domestic and agricultural activities and other geological, environmental and global changes [3]. Nowadays, water pollution is a serious issue because it affects our lives and is expected to get worse over coming decades. More than seven hundred organic and inorganic pollutants have been reported in water along with microbial populations [4]. Various methods for water purification and recycling have been developed and used [5-6]. The most important are reverse osmosis, ion exchange, electrodialysis, electrolysis and adsorption. Among these, reverse osmosis, ion exchange, electrodialysis and electrolysis are costly technologies with a 10–450 US$ per million liter cost for treated water. The cost of treated water by adsorption varies from 10 to 200 US$ per million liters. Adsorption is a fast, inexpensive and widely applicable technique [7]. Moreover, it is universal in nature as it can be applied for the removal of soluble and insoluble contaminants and biological pollutants with removal efficiency of 90–99%. At an industrial level, pollutants are

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removed from water by using columns and contractors filled with suitable adsorbents. Adsorption can also be used for source reduction, reclamation for potable, industrial and other purposes. As a result, much work has been carried out on water treatment by adsorption [8].

Enteric viruses have been detected in waters determined compliant for bacterial indicators, and subsequently linked to viral-associated outbreaks [9].

The development of detection techniques based on molecular biology has enabled us to detect fragments of viral genomes in environmental waters, including drinking water sources, highlighting the need to ensure the removal of viruses at drinking water treatment plants. Although disinfecting water with hypochlorite ensures the biological safety of the finished water, the risk of virus infections can be reduced by physicochemical treatments such as coagulation–sedimentation–sand filtration; physical sieving processes such as ultrafiltration, nanofiltration, and reverse osmosis; and ozonation and UV irradiation [10].

Activated carbon is extensively used as efficient and versatile adsorbents for purification of water, air and many chemicals and natural products. The application of high-surface active carbons in gas separation, medicine and catalysis is also well known. The continuously increasing list of environmental concerns and the interest in utilization of various wastes have awaken the interest for development of new processes for production of carbon adsorbents based on agricultural and forest wastes. Activated carbon produced from coconut shells and pine wood wastes has shown good mechanical strength and high adsorption capacities towards various gases. Olive stones and date stone are also suitable raw materials for activated carbon with high adsorption capacities, sufficient mechanical strength, and low ash contents [11].

The present study was designed to examine the sorption of viruses on Active carbon prepared from olive and date stones in greater detail using controlled laboratory conditions. Effects of various parameters on sorption were also studied so as to be able to delineate the sorption mechanism.

2. Materials and Methods

2.1. Chemicals

Zinc Chloride (ZnCl2), Hydrochloric acid 37 % (HCl), Methylene blue (C16H18ClN3S,xH2O), Sodium bicarbonate (NaHCO3), Calcium carbonate (CaCO3), Calcium chloride (CaCl2), Xylene (C8H10), sodium thiosulphate pent hydrate (Na2S2O3,5H2O), Iodine (I2), Potassium iodide (KI), starch were obtained from Alfa Chemical Group. All the chemicals were of high purity (99.999%) and utilized without further refining.

2.2. Raw Material

Stones were obtained from a packaging factory in 10th Ramadan City. Both washed thoroughly with water to remove all foreign materials, then, they were spread in one layer over plastic sheets and left to dry indoors. Washed-clean whole then dried in a drying oven at 110oC to facilitate crushing and grinding.

2.3. Preparation of the activated carbon

The dried stones were soaked in ZnCl2 solution (2.0 g/g) for 24 h at 110 °C. The dried mixture were put into a furnace and heated for (120 min) at temperature (700°C) under a constant N2 (99.99%) flow of 120 cm3/min. Then, the produced activated carbon was repeatedly washed with 0.1 Mol/L HCl followed by hot distilled water. Finally, the product was dried at 110 °C for 24 h, ground and sieved to a particle size for further studies [12].

2.4. Characteristics of prepared activated carbon at optimum condition

2.4.1. Bulk density

Bulk or apparent density is a measure of the weight of material that can be contained in a given volume under specified conditions. A 10 ml cylinder was filled to a specified volume with activated carbon that had been dried in an oven at 80 ˚C for 24 h [13]. The bulk density was then calculated as follows:

Bulk density = Wc / Vc

2.4.2. Ash content

The total ash content of the materials was determined using crucibles in a muffle furnace at 600 °C. After heating, the crucibles were allowed to cool in a desiccator and weighed [14]. The residue weight was calculated and reported as percentage of ash by

$$\text{Ash (\%)} = \frac{Ws3 - Ws2}{Ws1} \times 100$$

2.4.3. Moisture content

The moisture content of the as-received materials was determined on oven-drying the material at 110° C until consistency of weight was obtained [15].

2.4.4. pH measurement

The pH value of prepared activated carbon was determined by immersing 1 g sample in 100 ml

deionized water and stirring at 150 rpm for 1 h and the pH of slurry taken [16].

2.4.5. Volatile matter determination

Samples were oven-dried at 110 C for 6 h to eliminate residual moisture. The oven dried sample was taken in a clean pre-weighed silica crucible and heated in a muffle furnace at 550 oC. The crucible cooled and weighed to determine the volatile solids or loss on ignition. The loss on ignition is the resulting percentage loss in mass of each sample [17].

2.4.6. Iodine number

Iodine number is defined as the milligrams of iodine adsorbed by one gram of activated carbon. Basically, iodine number is a measure of the micropore content of activated carbon (0 to 20 Å) by adsorption of iodine from solution. The iodine number was then calculated by using the equation [13].

2.4.7. Methylene blue number

The methylene blue number is defined as the maximum amount of dye adsorbed on 1.0 g of adsorbent. In this assay, 10.0 mg of activated carbon are placed in contact with 10.0 mL of a methylene blue solution at different concentrations (10, 25, 50, 100, 250, 500 and 1000 mg L-1) for 24 h at room temperature (approximately 25°C). The remaining concentration of methylene blue is analyzed using a UV/V is spectrophotometer (Mindray BA-88A) at 645 nm. [18].

2.4.8. Morphology of activated carbon

This test method covers the testing of interior surfaces of components such as tubing, fittings, and valves for surface morphology, using Scanning electron microscopy (ZEISS model EVO 10 SEM) [19].

2.4.9. Specific surface area

Nitrogen adsorption experiments at 77 K were conducted to determine the specific surface area of the test samples using a Quanta chrome Nova-1200 instrument. The samples were outgassed overnight at 180 °C prior to adsorption measurements. The BET model was applied to the nitrogen adsorption isotherms and evaluates the specific surface area of the samples [20].

2.5. Virus preparation

Bacteriophage against Acinetobacter baumannii was selected as the model virus for this study because of its stability in agitated systems and its greater ease of culturing and enumeration. Bacterial and animal viruses have many similar physical, chemical and biological properties, i.e. size. net electric charge, protein coat, etc. Infected cells and fluids were frozen at -20°C and thawed thrice. Clarified supernatant after centrifugation at 1800g for 30 rain was filtered through presoaked membrane filter (ǿ= 0.2 μm) in 3% beef extract to obtain mono dispersed virus stock suspension. The filtrate which had a titer of 90*107 PFU ml (virus stock) was then distributed into 5 ml quantities and stored at -20°C. [10] Virus concentrations were measured by the plaque-forming unit (PFU) method according to the agar overlay method [21].

2.6. Column test

Column systems containing equal weights (25.8 g) of our compared active carbons to assess how each carbon types would perform as a single-pass, point of-use device. A flow rate of 4.33 mL/min of virus-solution was established with a peristaltic pump and passed through columns of 1.9 cm diameter and length 50 cm with three outputs at 15 cm, 30cm and 45cm glass fiber filters were used on all ends to hold granular 30 × 40 mesh active carbon in place. Sterile tubing was used to connect the columns, pump, and fluid reservoir together. Using three concentrations (24*104, 29*105 and 36*105) PFU/mL [22].

2.7. Batch adsorption test

Comparative studies were performed with three types of active carbon two types that we prepared and commercial one (Oxford). Deionized water was buffered with 424 μM NaHCO3 to give the equivalent of 20 mg-CaCO3/L of alkalinity. The buffered water was supplemented with 500 μM CaCl2. In a square beaker, 500 mL of solution was adjusted to pH 6.8 with HCl, and Bacteriophage was added to give three concentrations (74*106, 52*105 and 36*104) PFU/mL. Powder Active carbon was added at 20 mg/L and the suspension was continuously stirred at G = 200 s 1 with a jar tester. Samples were withdrawn at 1/2, 1, 11/2, 2, and 3 h and filtered through a membrane (ǿ = 0.2 μm) to remove the Powder Active carbon particles. The virus concentration in the permeate was measured by the PFU method [10].

2.8. Electrophoretic mobility

Batch test repeated while the buffered water was supplemented with seven concentrations of CaCl2 (0, 100, 200, 300, 400, or 500 μM). All solutions were

held for 1 day at 20oC for the pH to stabilize. Just before measurement. The electrophoretic mobility of powder active carbon and viruses was measured with an electrophoretic light-scattering spectro-photometer (Zetasizer Nano ZS, 532 nm green laser; Malvern Instruments Ltd., Malvern, Worcestershire, UK) at 25 C and at a 17 measurement angle [10].

2.9. Virus hydrophobicity

Hydrophobicity was estimated by the bacterial adhesion to hydrocarbon (BATH) method. Virus was added to 3 mL of buffered water at a final concentration of 25*106 PFU/mL at pH 7.0. The solution was supplemented with 0.25 mL of solvent (p-xylene). The solution was intensely vortexed for 2 min, and then rested for 15 min at room temperature to allow the solvent and water to separate. The virus concentration in the water phase was measured by A bicinichoninic acid assay (BCA) quantifies virus by measurement of the total amount of protein in sample. A decrease in virus concentration was used as a measure of the virus surface hydrophobicity [23].

3. Results and discussion

In addition to batch adsorption isotherm and column breakthrough studies were conducted to compare two types of activated carbon with very different structural characteristics.

3.1 Physico-chemical characteristics of activated carbon prepared from olive and date stones

In order to optimize an activated carbon preparation studied for proximate analysis. pH value, moisture content, ash content, bulk density, iodine number, methylene blue number, and specific surface area.

Table (1): Physico-chemical characteristics of activated carbon derived from olive and date stones:

Parameter	Olive	Date
pH	5.9	5.3
Moisture %	0.5	0.8
Ash %	25	19.6
Water soluble material %	1.57	1.8
Acid soluble material %	2.08	2.3
Volatile mater %	24	27
Iodine value (mg/g)	848	768
Methylene blue absorption (mg/g)	182	148
Bulk density (g/cm3)	0.64	0.85

3.1.1 pH

The determined value of pH for the activated carbon prepared from olive and date stones were 5.9 and 5.3, respectively. Distilled water was used for washing the carbonized sample with an aim to remove ZnCl2 solution which was used to chemically activate the carbon. Since the pH of distilled water was in the range of 5 to 6, hence the pH indicates the washing out of ZnCl2 solution from the sample [24].

3.1.2 Moisture Content

Moisture content dilutes the carbon and increases the weight during treatment process. Thus, the lower the moisture contents in activated carbons, the better [25].The moisture content for activated carbon was as low as 2 %, indicating a good carbon. With a slight preference for Olive.

3.1.3 Ash Content

The low ash content value for activated carbon (19.6 %) derived from date a stone indicates that have low inorganic content and high fixed carbon than activated carbon derived from olive stones. This is because ash content can reduce the efficiency of reactivation. Therefore, the lower the ash content, the better the activated carbon [25].

3.1.4 Bulk Density

It can be seen that the bulk density for olive stones s activated carbon is 0.64 g/cm3 and 0.85 for date stones. Bulk density determines the mass of carbon which can be contained in a filter by a given solids capacity. It also determines the amount of treated liquid that can be retained by the filter cake [24].

3.1.5 Iodine number

Iodine number is a measure of the micropore content of the activated carbon. The iodine molecule is relatively small with an area [26] of 0.4 nm2 and can enter in the smaller micropores [27]. In addition, the iodine value olive stones activated carbon is (848 mg/g) shows high micropore content in the activated carbon than derived from date stones which give (768 mg/g).

3.1.6 Methylene blue number

Methylene blue number obtained olive stones activated carbon is 182 mg/g. better than activated carbon than derived from Date stones. From the literature methylene blue number increase found to be the best represented activated carbon by this model [28], Adsorption of basic dye (methylene blue) onto activated carbon prepared from rattan sawdust, dyes & pigments, 75, 143-149.

3.1.7 Water soluble material

Water soluble material value for activated carbon from date is 1.8 % was higher than activated carbon from olive that was 1.57 %.

3.1.8 Acid soluble material

Acid soluble material value for activated carbon from date is 2.3% was higher than activated carbon from olive that was 2.08% for activated carbon from Date is1.8% was higher than activated carbon from olive that was 1.57 %.

3.2 Characterization of activated carbon bet surface area and pores volume

The BET method was used for the determination of the total pores surface area and volume. Thereby, the volume of the pores in the activated carbon obtained from the BET method could be used to estimate the surface area and volume of the micropores. From table 2, it can be observed that both surface area, microspore volume and micropore area of the activated carbon prepared from olive more than date. While total pore volume, total pore diameter and average pore diameter of the activated carbon prepared from olive less than date. This increasing can be due to the volatilization of melted ashes that results in the opening of the blocked micropore channels, which results in the slightly larger amount of micropores.

Table (2): Detailed characteristics of surface area with the carbons:

Parameter	Olive	Date
Surface Area (m^2/g)	931.679	203.240
Total pore volume (cc /g)	0.089	0.130
Total pore diameter (nm)	1.677	3.651
Average pore diameter (nm)	1.65516e+00	3.75431e+00
Micropore volume (cc /g)	0.335	0.054
Micropore area (m^2/g)	650.913	112.269

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3.3 Surface Morphology
Structural analysis by Scan Electron Microscope (SEM)

Using microscopy, it is possible to observe the micro and submicro-features of activated carbons directly and therefore makes possible a proper qualitative and quantitative description of its characteristics. The SEM is one version of the electron microscopy which uses a beam of electrons to scan the surface of a specimen and makes possible the direct observation of its surface features at the micro and submicro levels.

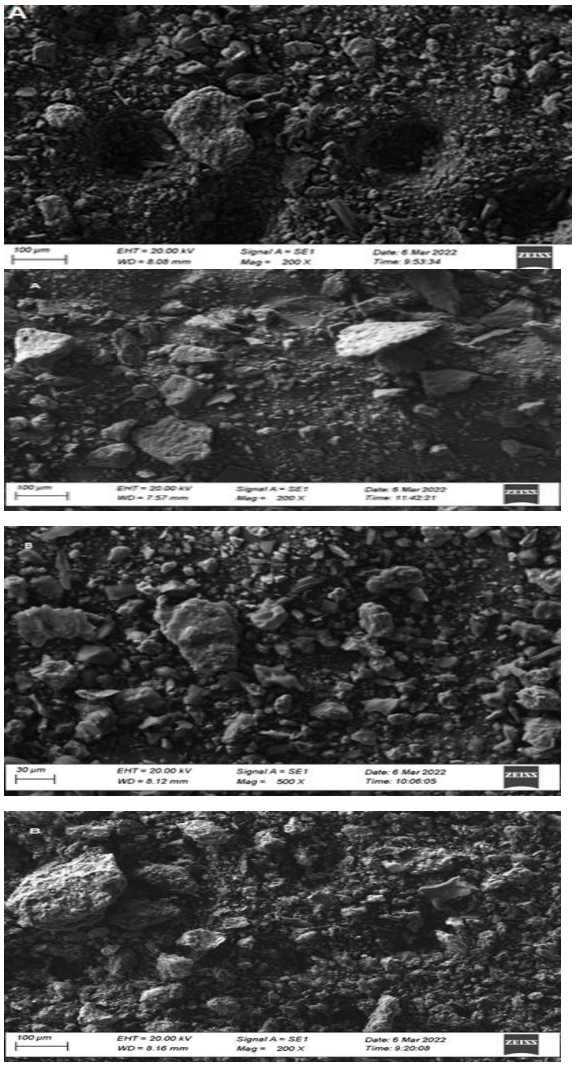

Figure 1: (A) SEM image of dates stones, (B) SEM image of olive stones.

Due to the huge magnifications and impressive resolutions achievable.

The scanning electron microscope was used to study the crystal structure and the pore development in activated carbon samples. SEM was focused on many directions to find the most representative images of the samples.

3.4. Column test

The influent and effluent concentrations were not significantly different so This study showed that granular active carbon (GAC) absorption filters are not good barrier in treatment for the elimination of viruses.

The preferred explanation is that Acinetobacter baumannii phage has very small diameter less than (ǿ= 0.2 µm) when compared with pores diameter on GAC surface.

Authors have reported a low capacity of GAC to eliminate viruses and bacteriophages [30-31-32].

Table (3): Comparison between the three types of prepared activated carbon by Column test to eliminate Phage after passage:

Initial titer of phage (Input) = 29×105 PFU/ml						
Distance	**Olive**		**Date**		**Commercial**	
	phage Output	**% of phage adsorbd**	**Phage Out-put**	**% of phage adsorbd**	**phage Output**	**% of phage adsorbed**
15cm	23×105	20.7	24×105	17.3	25×105	13.8
30cm	21×105	27.6	22.9×105	21.1	23×105	20.7
45cm	20.3×105	30.0	20.8×105	28.3	21.7×105	25.2

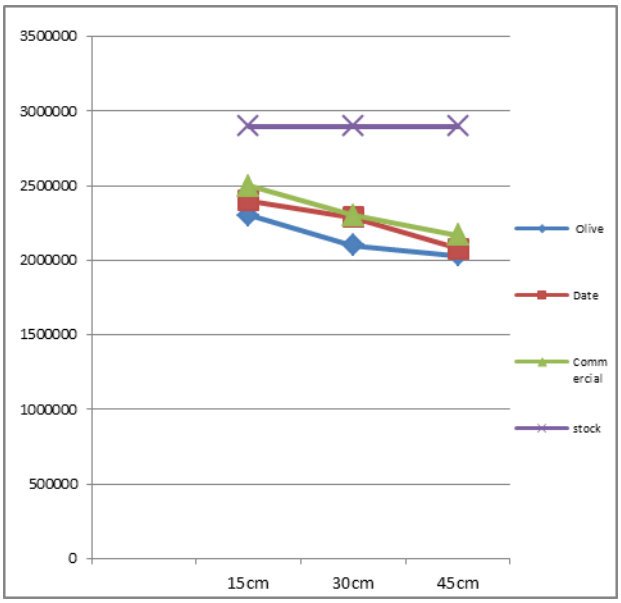

Figure (2): Comparison between the three types of prepared activated carbon by column test to eliminate phage after passage.

3.5. Comparison of virus removal by batch adsorption test

Virus removal increased with time in all types of PAC as showed in table (4) but the two prepared PAC from olive and date give better results than commercial by repeated tests with different phage stokes.

Table (4): Comparison between the three types of prepared activated carbon by batch adsorption test to eliminate Phage after passage:

Initial titer of phage (Input) = 52×105 PFU/ml						
Time	Olive		Date		Commercial	
	Phage Output	% of phage adsorbed	Phage Output	% of phage adsorbed	Phage Output	% of phage adsorbed
30 min	26×105	50	33×105	36.6	36×105	30.8
60 min	16×105	69.7	19×105	63.5	23×105	55.8
90 min	14×105	73.1	8×105	84.7	16×105	69.3
120 min	7×105	86.6	4×105	82.4	11×105	78.9
150 min	2×105	96.1	3×105	94.3	8×105	84.7

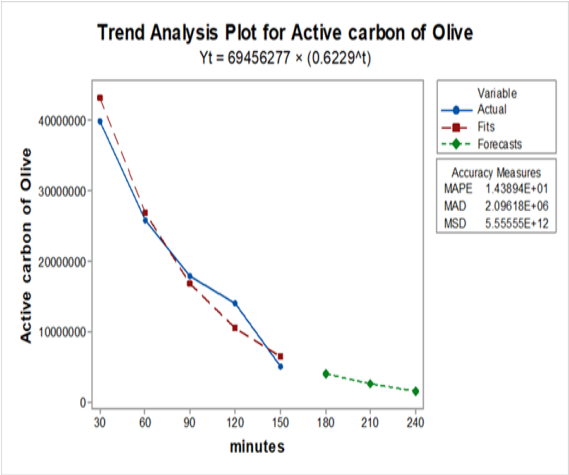

Figure (3): Virus removal by batch adsorption test by active carbon of olive.

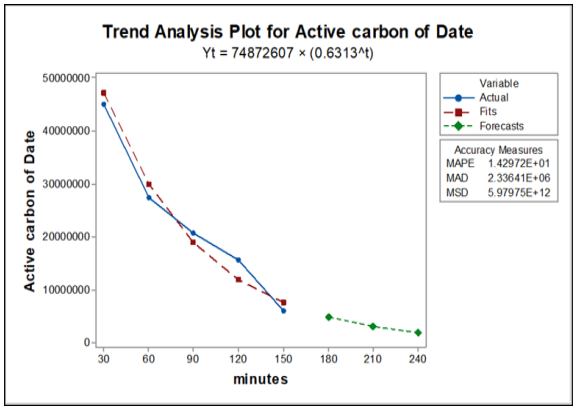

Figure (4): Virus removal by batch adsorption test by active carbon of date.

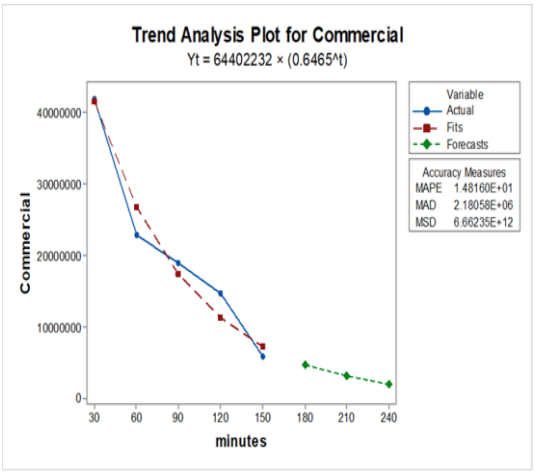

Figure (5): Virus removal by batch adsorption test by commercial active carbon.

By the Trend Analysis of results, we found that the amount of virus adsorbed had reached (>99% removal) as in figures (3, 4, 5) we monitored the concentration of virus in the liquid phase, which might have decreased further even after 99% of the virus was adsorbed.

Virus removal may be influenced by many factors such as raw material, specific surface area, element content, surface functional group, pore size distribution and surface charge.
Never the less, as the capacity of the olive-based PAC not varied widely by fitted trend equation, and a date-based PAC removed more virus than some commercial -based PAC, the difference in virus removal seems to be due to more than the raw materials.

The inherent characteristics of PAC listed in table 1, i.e. specific surface area and ash content.

3.6. Effect of surface charge using electrophoretic mobility:

When virus and PACs were dispersed in water without Ca^{2+}, both particles were highly negatively charged and the electrostatic repulsive force between them, measured as electrophoretic mobility.

Table (5) as the Ca^{2+} concentration increased the electrophoretic mobilities of both virus and PAC decreased. In general, an increase in ionic strength compresses the diffuse layer of ions surrounding a charged particle, decreasing the extent of the charge. This behavior has been seen before in viruses [33].

Table (5): Effect of surface charge Using electrophoretic mobility:

S No.	Ca^{+2} conc. µ.	Repulsion force 10^{-20} J
1	0	14
2	0.1	12
3	0.2	10
4	0.3	8
5	0.4	6
6	0.5	4
7	0.6	2

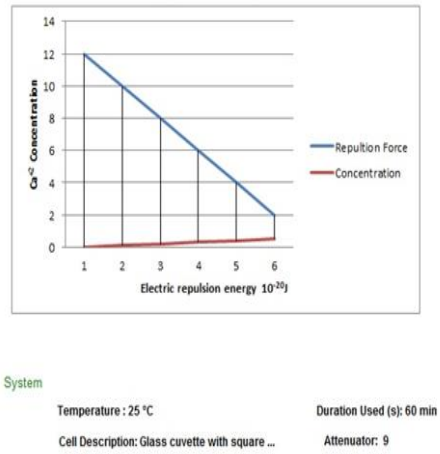

Figure (6): Effect of surface charge using electrophoretic mobility.

When virus and PAC were spaced reduced, as the Ca^{2+} concentration increased, the repulsion decreased figure (6). Repulsive forces between colloids of like charges can be lowered by decreasing the distance of charge effectiveness, a function of the ionic strength [34] Therefore, reducing the repulsion by increasing the ionic strength improved virus removal. One explanation is that the positive ions shield the negative charges on the surfaces of the adsorbate and the adsorbent, decreasing the net electrostatic repulsion between the particles. Or Ca^{2+} may electrically adsorb to a negatively charged moiety of both adsorbate and adsorbent concurrently, forming a cation bridge to link the like-charged particles.

3.7. Effect of hydrophobicity of virus on removal

A simple quantitative method has been described for studying the outer cell surface of bacteriophage, based on the affinity of these cells for liquid hydrocarbons. Bacteriophage remained in the water phase of xylene solvent, indicating that it has a hydrophilic surface.

Results illustrating the adherence of bacteriophage to the test hydrocarbons are presented in table (6)

Table (6): Effect of surface charge using electrophoretic mobility

Xylene solvent volume (μm)	Absorbance
100	0.508
200	0.354
300	0.190
400	0.070

There is an inverse relationship between absorbance and xylene volume. Decrease in absorbance of the aqueous phase was used as a measure of cell surface hydrophobicity. Thus, the more hydrophobic the surface of the virus particles is, the greater the virus removal would be expected. Reducing the surface charge of the activated carbons improved virus removal.

The reduction in the surface charge may provide more hydrophobic surface on the carbon apparently, because the reduction allows negatively charged adsorbents to move nearer to the graphite structure on the carbon [10]. Likewise, the hydrophobicity of the viruses contributed to the high removal: the virus having more hydrophobic surface was removed more greatly with the activated carbons.

4. Conclusion

Based on the laboratory investigations reported, using a bacterial virus, the following conclusions may be drawn.

1) The value-added activated carbon of date and olive stones can be produced by simple carbonization-chemical activation.
2) The characterization of the resulting activated carbon shows excellent physical and chemical properties suitable to be used as adsorbent.
3) Electrophoretic repulsive force and the pore size contributed greatly to virus removal.
4) Super-powdered activated carbon prepared from date and olive stones successfully removed buffered the virus in water than commercial active carbon.
5) Granular activated carbon has not been a recognized method to remove waterborne viruses.
6) The hydrophobicity of the virus play an important role in virus removal: the more hydrophobic the surface, the greater the virus removal.

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