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Adsorption of Toxic Methyl Violet 2B Dye from Aqueous Solution Using Artocarpus heterophyllus (Jackfruit) Seed as an Adsorbent

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Authors' contributions

This work was carried out in collaboration between all authors. Author MKD performed the experiments and wrote the first draft of the manuscript. Author MRRK performed the experiments and did the analysis of data. Author LBLL improved the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

The potential of Artocarpus heterophyllus (jackfruit) seed (JS) as an adsorbent to remove methyl violet 2B (MV) dye was investigated using batch experiment method. Surface morphology and functional groups of JS were analyzed in order to characterize the adsorbent. The adsorption experiments were carried out by varying the pH, dye concentration, contact time, ionic strength and temperature. Adsorption isotherm, kinetics and thermodynamics were also studied to further understand the adsorption process. Isotherm data was described using three isotherm models of which the Langmuir model best represent the experimental data with maximum adsorption capacity of 126.7 mg g^{-1} . Pseudo 2nd order described the adsorption mechanism and intraparticle diffusion was not the rate limiting step. Regeneration studies showed that JS was able to maintain good adsorption of MV dye even after a few cycles.

electrostatic interaction; adsorption.

Keywords: Artocarpus heterophyllus (jackfruit) seed; methyl violet dye; water remediation;

1. INTRODUCTION

About 25% of today's diseases that afflict humans are due to the exposure to environmental pollution whether from air, land or water for a period of time and this has become a global concern [1]. Pollution continues to rise since the start of industrial era as a result of waste products that are usually thrown away in water bodies. Amongst these waste products are synthetic dyes which are widely used in textile, paper, cosmetic and food industries and are disposed in huge amount every year. Improper disposal of these dyes would cause harm to flora and fauna in rivers, lake and sea as they can hamper photosynthesis and can pose human health risk as they are potentially carcinogenic [2]. Synthetic dyes are chemically stable and resistant to biodegradation and thus, will persist in the water bodies for a long period of time if ignored [3].

Various methods have been used to treat the dye effluent wastewater [4]. However, most of these methods are usually expensive, inefficient and might produce unwanted by-products. Adsorption has also been applied in water remediation which produced satisfactory results. The common material used in adsorption is activated carbon but recent studies have shown that other materials such as soil materials [5], fruit peel [6], weed [7], agricultural wastes [8], and others [9-12] can also be used as adsorbents. These materials are cheap, easy to find and abundant in nature which makes them attractive alternatives to activated carbon.

Methyl violet 2B (MV), a basic dye that belongs to triphenylmethane class, is a dark green solid and dissolves in water to give an intense violet color. The structure of MV is shown in Fig. 1. The dye has broad applications in paints, textile and printing ink. It is an active ingredient in biological stain and can also be used as a moderate-class disinfectant. However, MV is revealed to be toxic as it can cause severe skin, respiratory, gastrointestinal tract and eye irritations [13]. Due to its intense colour, even a small amount of MV in the water would produce a noticeable coloration. For these reasons, it is imperative to have an effective method to remove MV.

Different parts of Artocarpus spp. such as leaf, peel and core have been used in adsorption studies and they have proven to be effective in the removal of dyes and heavy metals [14-18]. The aim of this study is to use the seed of Artocarpus heterophyllus (jackfruit) as an adsorbent to remove MV dye from aqueous solution. To date, there has not been any report on the adsorption of MV onto JS. Jackfruit is a popular fruit that can be found in the South-East Asian nations, India, Australia, Africa, Caribbean islands and most of the Pacific islands [19]. The seed consists of 8-15% of the total fruit weight [20] which can be roasted, boiled or steamed to produce aromatic and nutty taste. This study can be perceived as an alternative use of jackfruit seed (JS).

Fig. 1. Molecular structure of MV

2. MATERIALS AND METHODS

2.1 Material and Chemical Reagents

Jackfruits were bought from local open markets and the seeds were separated from the fruit, diced, washed and dried in Gallenkamp Hotbox oven at 70ºC until a constant mass was obtained. The dried seeds were then blended using Panasonic MX-J210GN blender and sieved to obtain the desired particle size of 355 µm. The sample was stored in a sealed plastic bag until further use.

Methyl violet 2B, IUPAC name N-(4-
(bis(4(dimethylamino) phenyl) methylene) (bis(4(dimethylamino) phenyl) methylene) cyclohexa-2,5-dien-1-ylidene) methanaminium chloride, with molecular formula $C_{23}H_{26}N_3Cl$ and molecular weight 394 g mol $^{-1}$ was purchased from Sigma-Aldrich. 500 mg L^{-1} dye stock solution (1000 mL) was prepared by dissolving

appropriate amount of solid in double distilled water. Lower dye concentrations (20 – 400 mg L⁻¹) were prepared by serial dilution of the stock solution. All reagents were used without further purification and double distilled water was used throughout the experiment.

2.2 Characterization of JS

Functional groups on adsorbent's surface were identified using Fourier transform infrared (FTIR) (Shimadzu IR Prestige-21 spectrophotometer). Spectroscopy grade KBr was dried in an oven at 110ºC before use. Samples (0.002 g) were grinded with KBr (0.2 g) using mortar and pestle. Surface morphology analysis of JS was carried out by using a Tescan Vega XMU scanning electron microscope (SEM).

2.3 Batch Adsorption Procedures

Batch adsorption method was used to investigate the effects of contact time $(5 - 180 \text{ min})$, temperature (25 – 55°C), pH (2 – 10), dye concentration (20 – 500 mg L^{-1}) and ionic strength ($0 - 0.8$ NaCl mol L⁻¹) on the adsorption of MV onto JS.

The effect of adsorbent dosage was carried out using pH-unadjusted 100 mg L^{-1} MV dye, and the studied dosage ranged from 0.01 g to 0.06 g, and 0.03 g was found to be the optimum adsorbent dosage and was used for the rest of the experiments (data not shown for brevity).

The effect of contact time was conducted by adding JS (0.03 g) to 50, 100 and 200 mg L^{-1} MV solutions (20.0 mL) solution and the mixtures where agitated at 250 rpm. Aliquots from each MV solution were taken at five min interval for the first 30 min and subsequently, the aliquots were taken every 30 min.

JS (0.03 g) was mixed with 50 mg L^{-1} MV (20.0 mL) and the mixture was agitated at different temperature to investigate the effect of temperature.

The pH of 50 mg L^{-1} MV (20.0 mL) solutions were adjusted to pH range of $2 - 10$ before mixing with JS (0.03 g). The mixtures were then agitated at room temperature for 2 h at 250 rpm.

Different concentrations of MV (20 – 500 mg L^{-1}) were prepared from the stock solution. JS (0.03 g) was added to the MV solution and agitated at room temperature for 2 h at 250 rpm.

The effect of ionic strength was done by preparing 50 mg L^{-1} MV solution (20.0 mL) using different amounts of 1.0 mol L^{-1} NaCl. JS (0.03 g) was then added and the mixtures were then agitated at room temperature for 2 h at 250 rpm.

The MV content in the filtrate was then analyzed using an UV–visible spectrophotometer (Jenway 6320D spectrophotometer) at wavelength 584 nm.

The point of zero charge (p H_{pzc}) was done by mixing JS (0.04 g) with 0.1 mol L^{-1} KNO₃ solution (20.0 mL). Prior to the mixing, the salt solution pH was adjusted using 0.1 mol L^{-1} HNO₃ and 0.1 mol L^{-1} NaOH from 2.0 to 10.0. The mixture was then agitated for 24 h using a Stuart orbital shaker set at 250 rpm. The final pH of the solution was measured using Thermo-Scientific pH meter and the pH_{pzc} was determined from the plot of ∆pH (final pH - initial pH) vs initial pH.

The adsorption capacity, q_e (mg g⁻¹), of JS is calculated using Equation (1) as follows:

$$
q_e = \frac{(c_i - c_e)v}{m} \tag{1}
$$

where C_i is the initial MV concentration (mg L^{-1}), C_e is the MV concentration in the filtrate (mg L^{-1}), V is the volume of MV used (L) and m is the mass of JS (g).

The % removal of MV by JS is calculated according to Equation (2) as follows:

% removal =
$$
\frac{(c_i - c_e) \times 100 \text{ %}}{c_i}
$$
 (2)

2.4 Regeneration Experiments

Fresh JS (0.1 g) was agitated in 50 mg L^{-1} MV solution (66 mL) for 2 h and the JS-MV mixture was then filtered. The spent JS was then divided into two portions. One portion was washed using double distilled until no further desorption of MV observed. The second portion was agitated with 0.1 mol L^{-1} NaOH for 30 min before washing using double distilled water till the pH of the effluent was near neutral. Both portions were dried in the oven at 70°C overnight. This process was repeated for three cycles.

2.5 Error Function Analysis

Two error functions i.e. the *Chi*-square (χ^2) and sum of absolute error (EABS) were used in this

study in order to determine the suitability of kinetics and isotherm models in describing the experimental data. The equations for χ^2 and EABS are shown in Equations (3) and (4), respectively as follows:

EABS:
$$
\sum_{i=1}^{n} |q_{e(\exp)} - q_{e(cd)}|
$$
 (3)

$$
\chi^2: \sum_{i=1}^n \frac{(q_{e(\exp)} - q_{e(cal)})^2}{q_{e(\exp)}} \tag{4}
$$

where $q_{e(exp)}$ is the adsorption capacity value from the experiment while $q_{e(cal)}$ is the adsorption capacity calculated from the kinetics and isotherm models. n is the number of experimental data used and p is the number of parameters of the model.

3. RESULTS AND DISCUSSION

3.1 Characterizations of JS

JS surface morphology was done using SEM at 400x magnification to provide visual details and is shown in Fig. 2. The surface of the adsorbent was observed to be irregular and rough without any distinct shape, characteristic or orderly arrangement.

Fig. 2. SEM image of a JS particle at 400 x magnification

Fig. 3 shows the FTIR spectra of JS before and after adsorption of MV dye. The stretching band seen at 3409 cm^{-1} in the JS spectrum indicates the presence of hydroxyl and amino groups. The C—H stretch, N—H bending, phenyl and C—O— C bands are observed at 2931, 1631, 1414 and 1022 cm^{-1} , respectively. After treatment with MV, shifts of bands are observed for O—H and amino groups, N—H bending, phenyl and C—O—C to 3410, 1641, 1442 and 1016 cm^{-1} , respectively. This suggests the involvement of these functional groups in the adsorption of MV onto JS. The band at 1366 cm^{-1} observed in JS-MV spectrum indicates the C—N stretching for amine presence in MV which may suggest the loading of the dye onto JS.

Point of zero charge (pH_{pzc}) is the determination of pH at which the surface is neutral. It is a useful parameter as it can be used to predict the surface charge depending on the medium pH. The surface will be predominately positive in charge when the medium pH is lower that the pH_{pzc} value. Likewise, if the pH is higher than pH_{ozc} , the surface will be predominately negative in charge. These changes occur due to the protonation or deprotonation of the surface's functional groups such as carboxyl and amino groups. From the plot of ∆pH (final pH - initial pH) vs initial pH (figure not shown for brevity), the pH_{pzc} of JS was determined to be at pH 3.75.

3.2 Effects of pH and Ionic Strength

The parameters pH and ionic strength are important in adsorption studies because in real wastewater effluents, the pH and ionic strength of the wastewater vary depending on the industry's working parameters i.e. dyeing might require low pH and addition of salt for it to be effective. pH can affect the functional groups of the adsorbent as discussed previously while the ionic strength can affect both electrostatic and hydrophobic interactions where increasing ionic strength will suppress the electrostatic interaction and enhance hydrophobic interaction [21,22]. The effects of pH on the adsorption of 50 mg L^1 MV onto JS is shown in Fig. 4A. It can be seen that, with the exception of pH 2, the adsorption of MV is almost the same throughout the pH range (85 – 90%). Percentage removal at pH 2 is the lowest at around 66% which is contributed by the predominately positively charged surface of JS at this pH. This causes electrostatic repulsion between the surface and cationic MV dye and thus, the uptake of MV is suppressed.

As seen in Fig. 4B, addition of NaCl to the adsorbate-adsorbent mixture reduced the MV uptake by JS. Without the addition of the salt, JS was able to remove 81% of MV, while in the presence of 0.1 mol L^{-1} and 0.8 mol L^{-1} NaCl, the percentage removal was reduced to 61% and 58%, respectively. This observation is to be expected in a system where the adsorbate and adsorbent are electrostatically attracted to each other whereby the adsorption capacity is decreased when the ionic strength increases. On the other hand, in systems where the adsorbent and adsorbate repel each other, an increase in ionic strength will increase the adsorption capacity [23]. The surplus of positive ions from the salt will compete for the adsorbent's adsorption sites and the adsorbed ions will increase the electrostatic repulsion between the surface and the cationic dye, thereby decreasing the adsorption capacity of the adsorbent. It is noted that the uptake of MV by JS was not severely affected by the change of pH and ionic strength (23% decrease in 0.8 mol L^{-1} NaCl), suggesting that electrostatic interaction did not play a major role in the adsorption process. This gives JS an advantage to be applied in real wastewater remediation.

3.3 Effect of Contact Time and Kinetics Modeling

For an adsorption process to achieve equilibrium, it is important to let both the adsorbate and adsorbent to be in contact for a certain period of time. Different adsorbents require different contact time depending on their structural and chemical nature. Due to this, it is important for the contact time parameter to be investigated. Fig. 5A shows the effect of contact time of different MV concentrations on the adsorption process. A general trend was observed throughout all the three MV concentrations used, whereby the q_e values sharply increased

between 5 to 30 min. Beyond 30 min, the adsorption slowed down and finally reached a plateau where no significant increase in q_e value was observed. This trend has been observed in other studies on MV by different adsorbents [24,25].

In order to study the adsorption mechanism, the pseudo $1st$ order [26], pseudo $2nd$ order [27] and Weber-Morris intraparticle diffusion [28] were applied to the experimental data and their equations (5 to 7) are expressed as follows:

Pseudo 1st order:

$$
log (q_e - q_t) = log q_{e(cal)} - \frac{t}{2.303} k_1
$$
 (5)

Pseudo 2nd order:

$$
\frac{t}{q_t} = \frac{1}{q_{e(cat)}^2 k_2} + \frac{t}{q_e}
$$
 (6)

Weber-Morris intraparticle diffusion:

$$
q_t = k_3 t^{1/2} + C \tag{7}
$$

where q_e is the equilibrium adsorption capacity (mg g⁻¹), q_t is the adsorption capacity (mg g⁻¹) at time t (min), $q_{e(cal)}$ is the calculated adsorption capacity (mg g^{1}, k_1 is the pseudo 1st order rate constant (min⁻¹), k_2 is pseudo 2nd order rate constant (g mg⁻¹ min⁻¹), k_3 is the intraparticle diffusion rate constant (mg g^{-1} min^{-1/2}) and C is the intercept.

Fig. 3. FTIR spectra of (A) JS-MV and (B) untreated JS

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Fig. 4. Adsorption of MV onto JS under various (A) pH and (B) ionic strength

The pseudo $1st$ order and the pseudo $2nd$ order models were used in order to determine the best kinetics model to describe the experimental data. The best model would have the highest coefficient of determination (R^2) value and smallest error values. From Table 1, the pseudo 2nd order has both of these qualities and furthermore, its calculated q_e values, $q_{e(cal)}$, were in agreement with that of experimental q_e , $q_{e(exp)}$, values suggesting that the adsorption of MV onto JS followed the pseudo 2nd order kinetics model. The pseudo $1st$ order model is not suitable to describe the entire time range of the adsorption process and this is confirmed by the deviation of its $q_{e(cal)}$ from the $q_{e(exp)}$. For example, using 200 mg L^{14} MV, the $q_{e(cal)}$ for pseudo 1st and 2nd order was 39.4 mg g⁻¹ and 134.8 mg g⁻¹, respectively while the $q_{e(exp)}$ was 135.1 mg g⁻¹, a value closer to that of pseudo 2^{nd} order.

Generally, in an adsorption process, film diffusion, intraparticle diffusion and equilibrium phases can be found [29] which can usually be seen in the Weber-Morris plots. Fig. 5B showed only two linear lines which represent intraparticle diffusion (first linear line) and equilibrium phase (second linear line). Film diffusion is a fast phase which is usually not seen in the plot. Intraparticle diffusion is considered as rate limiting step if the linear line passes through the origin. As seen in Table 1, the intercepts are not zero for all the MV concentrations which indicate that intraparticle diffusion was not the rate limiting step for the adsorption of MV onto JS.

Fig. 5. (A) Effect of contact time on the adsorption of MV onto JS and (B) Weber-Morris intraparticle diffusion model plots

3.4 Effect of Various MV Concentration and Adsorption Isotherm Analysis

Fig. 6 shows the adsorption capacity of JS at different MV concentrations. A rapid increase in q_e value from 20 mg L⁻¹ (11 mg g⁻¹) to 200 mg L⁻¹ $(91 \text{ mg } g^{-1})$ was observed and this increase is contributed by the filling of JS's adsorption sites and also caused by the driving force provided by the concentration gradient. Beyond 200 mg L^{-1} , the adsorption slowed down to the point where

the q_e value did not change significantly. The saturation of JS's adsorption sites by MV molecule causes the slowing down of the adsorption process which eventually will reached an equilibrium state where the q_e value will no longer increase significantly.

In order to have a better understanding of the adsorption process, the Langmuir [30], Freundlich [31] and Sips [32] isotherm models were selected to fit into the experimental data. These models provide useful insight on how the adsorption occurs on the adsorbent as well as predicting the quantity of the adsorbate being adsorbed by the adsorbent. These isotherm models were discussed in the literature [33]. The Langmuir, Freundlich and Sips models' linear equations are given in Equations (8) to (10) as follows:

$$
Langmuir: \frac{c_e}{q_e} = \frac{1}{K_L q_m} + \frac{c_e}{q_m} \tag{8}
$$

$$
\text{Freundlich: } \ln q_e = \frac{1}{n_F} \ln C_e + \ln K_F \tag{9}
$$

$$
\text{Sips: } \ln\left(\frac{q_e}{q_m - q_e}\right) = K_{LF} \ln C_e + \ln K_S \tag{10}
$$

where q_m (mg g⁻¹) is the adsorbent's maximum adsorption capacity, K_L (L mg⁻¹) is the Langmuir constant, K_F (mg g⁻¹ (L mg⁻¹⁾¹⁷ⁿ) is the adsorption capacity of the adsorbent, n_F value (between 1 and 10) indicates favorability of the adsorption process, $K_S(L g^{-1})$ is Sips constant and K_{LF} is the exponent.

The dimensionless separation factor (R_l) can be calculated using the Langmuir model's constant K_l and this parameter is useful in predicting the favorability of the adsorption process [34]. R_L can be calculated using Equation (11) as shown below:

$$
R_L = \frac{1}{(1 + K_L C_i)}
$$
 (11)

When the R_l = zero, the adsorption is irreversible; unfavorable adsorption when R_{L} 1; linear adsorption when R_1 = 1; and the adsorption is considered favorable when $0 < R_L < 1$ [35].

	Pseudo 1 st order		
C_i (mg L^{-1})	50	100	200
$q_{e(cal)}$ (mg g ⁻¹)	7.7	19.1	39.4
$q_{e(exp)}$ (mg g ⁻¹)	29.8	53.3	135.1
k_1 (min ⁻¹)	0.019	0.015	0.012
R^2	0.833	0.965	0.974
χ^2	201.2	274.8	831.7
EABS	238.0	339.3	955.5
	Pseudo 2 nd order		
$q_{e (cal)}$ (mg g ⁻¹	30.2	54.1	134.8
$q_{e(exp)}$ (mg g ⁻¹) k_2 (g mg ⁻¹ min ⁻¹) R^2	29.8	53.3	135.1
	0.008	0.003	0.001
	0.999	0.999	0.997
χ^2	9.7	13.8	31.4
EABS	36.3	69.5	167.6
	Weber-Morris		
	intraparticle diffusion		
k_3 (mg g ⁻¹ min ^{-1/2})	1.610	3.849	9.048
С	16.344	22.613	70.889
\mathcal{R}^2	0.940	0.985	0.868

The best fit isotherm model with the experimental data was chosen based on the highest R^2 value as well as smallest values for both EABS and χ^2 error functions. Table 2 summarizes the

Fig. 6. The adsorption of different MV concentration onto JS

adsorption isotherm models' parameters. In terms of R^2 , the Langmuir model has the highest value followed by Sips and Freundlich and this is in agreement with both the error functions whereby the Langmuir has the smallest error values. This showed that the Langmuir model, which assumes that the adsorbate will only form one layer on the adsorbent's surface, can be used to describe the adsorption process. The maximum adsorption capacity (q_m) of JS is determined as 126.7 mg g^{-1} . This value is comparable to other studied adsorbents such as tarap peel (137.3 mg g^{-1}) [36], halloysite nanotube (113.6 mg g⁻¹) [37], peanut straw char $(101.0 \text{ mg g}^{-1})$ [38] and Posidonia oceanica (L.) leaf (119.1 mg g^{-1}) [9]. However when compared to Azolla pinnata (194.2 mg g^{-1}) [25], cempedak durian (238.5 mg g⁻¹) [39], duckweed (332.5 mg g^{-1}) [40] and water lettuce (267.6 mg g⁻¹) [24], JS has a lower adsorption capacity towards MV dye.

3.5 Thermodynamics Experiments

The effect of temperature on the adsorption of MV onto JS and thermodynamics parameters are summarised in Table 3. It can be said that temperature has little effect on the MV uptake by JS as the q_e value stayed almost constant throughout the temperature range i.e. 28 mg q at 25 \degree C and 27 mg g⁻¹ at 55 \degree C.

Thermodynamics parameters are calculated using Van't Hoff equation and are expressed as:

$$
\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \tag{12}
$$

$$
\Delta G^{\circ} = -RT \ln k \tag{13}
$$

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$$
k = \frac{c_s}{c_e} \tag{14}
$$

$$
C_s = C_i - C_e \tag{15}
$$

Inserting equation (12) into equation (13):

$$
\ln k = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \tag{16}
$$

where T is the temperature in Kelvin (K), ΔG° is the Gibbs' free energy, ΔS° is the change in entropy, ΔH° is the change in enthalpy, k is the distribution coefficient, C_s is the amount of dye adsorbed on the adsorbent (mg L^{-1}), and R is the gas constant $(8.314 \text{ J mol}^{-1} \text{ K}^{-1}).$

In terms of thermodynamics, the adsorption of MV onto JS was spontaneous and feasible as indicated by the negative values of ΔG° . Although temperature displayed small effect on the adsorption, positive value of the ΔH° indicates the system was endothermic while the positive value of ∆S° indicated an increase in randomness within the system.

3.6 Regeneration Experiments

Spent adsorbents should be treated as hazardous wastes due to the adsorbed dyes and the dyes can leach back into the environment if dispose by landfill. The proper way to dispose hazardous dye waste is by incineration, however the fuel will increase the total cost of waste treatment. Regeneration of the spent adsorbent allows the adsorbent to be reused thereby

Fig. 7. Regeneration of spent JS using distilled water and 0.1 mol L-1 NaOH

providing alternative way to incineration. There are many methods of regeneration which can be done by physical, chemical of thermal methods. In this study, distilled water and 0.1 mol L^{-1} NaOH were used as they are easily available and does not require any complex machinery to perform. As seen in above Fig. 7, the adsorption capacity of JS was regenerated satisfactorily by both of the washing solutions throughout three cycles. The uptake of 50 mg L^{-1} MV was initially 81% and after three cycles both distilled water and base managed to remove 75% and 86% of MV, respectively. This is considered a great advantage especially when distilled water was sufficient to be used in the regeneration of JS.

4. CONCLUSION

The adsorption of MV onto JS was conducted at 2 h without any pH alteration. It was found that temperature has little effect on the adsorption process while ionic strength suppressed the adsorption capacity only by 23% in the presence of 0.8 mol L^{-1} NaCl. The adsorption process followed the pseudo $2nd$ order kinetics and the Langmuir isotherm model best fitted the experimental data with a maximum adsorption capacity of 126.7 mg g^{-1} . Thermodynamics study showed that the process was endothermic and spontaneous in nature. JS has a great advantage where it can be satisfactorily regenerated using both distilled water and base.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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