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Synthesis of Polyaniline for Water Remediation and Evaluating Its Feasibility to be Reused as Electrical Conductor

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ABSTRACT

Adsorption is an effective process for humic acid removal; however, disposing the spent adsorbents creates secondary waste to the environment. Hence, this study aims to investigate the feasibility of using polyaniline (PANI, a conductive polymer) to adsorb humic acid. Later on, possibility to recycle the spent PANI as electrical conductor is evaluated. The PANI was found to be 352.96 nm in size and exhibits the N-H functional group. Application study showed that the adsorption efficiency increased from 49.3% to 89.5% when the initial concentration of humic acid increased from 50 mg/L to 200 mg/L. Meanwhile, the increase in the PANI dosage successfully enhances humic acid removal owing to the existence of more adsorbing sites. The spent PANI was then deposited onto a flexible sheet and tested for its electrical conductance. Interestingly, the spent PANI-deposited sheet was found to exhibit lesser conductance (6.94×10^{-5} S) than the fresh PANI-deposited sheet (1.03×10^{-3} S). Also, the magnitude of electrical conductance further reduced with the increase in the amount of humic acid adsorbed onto the PANI. Apparently, the adsorbed humic acid has shielded the electron movement. It is believed that this study will provide new insight on the sustainable usage of water treatment-adsorbents.

Keywords: Electrical Conductance, Humic Acid, Polyaniline, Sustainability, Water Treatment.

1. INTRODUCTION

Humic acid is a type of dissolved organic matter which is normally found in rivers, pond water or lakes. It comes from the soil and formed through the chemical and biological humification of plants and animals. Despite being formed naturally, the presence of humic acid in the water resources is somehow undesired as it deteriorates the water quality. It turns the water colour to yellowish or brownish, stimulates bacterial growth [1-4], as well as induces the formation of trihalomethane (THM) in chlorinated-water [5, 6]. More importantly, humic acid contains various functional groups that can form complexes with heavy metal ions [7, 8] and thus keeps them inside the water resources. Realizing these adverse impacts, significant attention has been allocated to remove humic acid from water resources.

Strategies to remove humic acid have been developed over the past few decades and this include the membrane filtration method [9], electro-coagulation method [10, 11], coagulation–flocculation method [12, 13], photocatalysis and oxidation method [14], as well as the adsorption method [15-17]. Among all the existing methods, adsorption has been considered as the most effective owing to its simple operations, easy to implement, as well as cost effectiveness. Activated carbon, biochar, and bentonite, kaolinite, and nanoparticles are some of

the common adsorbents employed for this purpose. Nevertheless, one of the concerns associated with this method is that the spent adsorbents must be dumped at the end of their application, especially, when the adsorbents no longer can be regenerated. Such process eventually creates a secondary pollution to the environment. Hence, research shall venture into the utilization of adsorbents which can be reused for other.

In view of this, the present study aims to use polyaniline (PANI), a conductive polymer, as the adsorbent for humic acid removal. Later, the feasibility to reuse the spent PANI for fabrication of flexible electrical conductor will be tested. PANI is feasible for pollutant removal owing to its well-defined electrochemistry and good environmental stability. It can be easily synthesized via a low-cost oxidation method [18, 19]. More importantly, PANI was found to be positively charged upon protonation of the N-H functional groups. Henceforth, it is feasible to electrostatically attract anionic pollutants. For instance, Li et al. reported that orange G, an anionic dye, is completely adsorbed by PANI in 90 minutes duration with the initial dye concentration, PANI dosage, and medium pH set at 100 mg/L, 0.05 g, and pH 5, respectively [20].

Beside serving as an adsorbent, PANI is also known as a conductive polymer. This electrically conductive property is ascribed to its conjugated π-bond system (*i.e.* overlapping of carbon *p* orbitals and alternating C-C bond lengths) [21]. This conjugated system contains delocalized electrons that are freely mobile throughout the molecule; hence, making PANI an electrical conducting polymer. The resistivity of PANI is normally measured in the temperature range from 80 K to 300 K and it is reported that the resistivity decreases as the temperature increases [22-24]. At room temperature, the electrical conductivity of pure PANI was reported to be at around 5.15 S/cm and it increases as the temperature increases [24]. Interestingly, PANI can be further coated onto solid substrate and make it conductive. In fact, the incorporation of PANI onto different substrates such as metals [25], wood pulp fiber [26], glasses [27, 28], textiles [29, 30], and nylon [31] has been a successful project reported by researchers. The coating can be done in many ways such as through direct polymerization of PANI onto the substrate, inkjet-printing by putting the PANI dispersion onto the substrate, SILAR method, and etc.

Despite the various applications mentioned above, there is no study revealing feasibility to reuse the PANI (which was already spent on water pollutant removal) for fabrication of flexible conductive sheet. Hence, the present work aims to adsorb humic acid from aqueous solution using PANI. The spent PANI will be later coated onto a flexible sheet and tested for its electrical conductance. The electrical conductance of this spent PANI sheet will be compared to a fresh PANI sheet.

2. MATERIAL AND METHODS

2.1 Materials

Aniline (≥ 99.5 %; Sigma–Aldrich, Malaysia), hydrochloric acid (aqueous, 37 %; J. T. Baker, Malaysia), ammonium persulfate (≥ 98 % ; Sigma–Aldrich, Malaysia), sodium hydroxide (≥ 97 %; Sigma–Aldrich, Malaysia), humic acid (technical grade; Sigma–Aldrich, Malaysia) were used as supplied. Meanwhile, distilled water was used in all solution preparation.

2.2 Synthesis of Polyaniline (PANI)

PANI particles were synthesized via the well-known oxidative polymerization method [32, 33]. Here, 9 mL of Aniline is prepared in a flat bottom flask and further diluted with 150 mL of distilled water. The flask was kept inside an ice bath to maintain the temperature range within 0°C to 5°C. On the other side, 0.5 molarity of ammonium persulphate solution was prepared by

adding 11 g of ammonium persulphate in a 100 mL of distilled water. This ammonium persulphate solution was then added into the aniline solution. Lastly, 10 mL of hydrochloric acid (9M) was dropwise added into the flask using a burette. The mixture was then stirred for 2 hours.

The formed PANI needed to be purified from excess reactants. In this regard, the PANI dispersion was centrifuged at 3500 rpm for a duration of 12 mins. The supernatant was decanted while the precipitate, which is the PANI, was washed with distilled water. The same steps were repeated for at least three times to ensure complete purification.

2.3 Preparation of Humic Acid

A stock solution of humic acid was prepared by dissolving 0.125 g of humic acid powder in 250 mL of distilled water. To assist the dissolution, few drops of 1M NaOH was *pre*-added to dissolve the powder before the addition of distilled water. The mixture was then left for 3 hours stirring at a constant speed of 700 rpm. Later on, the formed humic acid solution was adjusted to pH 8 – 8.3.

2.4 Adsorption of Humic Acid: Effect of Humic Acid Concentration

In brief, 1.75 g of PANI was added to 5 mL of 50 mg/L humic acid solution for the batch adsorption study. The mixture was then centrifuged, and the supernatant obtained was analysed using UV-visible spectrometry at a wavelength 350 nm. Calibration curve was developed for humic acid at this specific wavelength and was found fitted to linear trendline with $R^2 > 0.98$. The obtained absorbance value was converted to concentration based on the generated calibration curve. Then, humic acid removal efficiency can be determined through Eq. (1),

$$\text{Humic acid removal \%} = \frac{\text{Initial concentration} - \text{supernatant concentration}}{\text{initial concentration}} \times 100 \% \quad (1)$$

The same procedure was repeated for 100 mg/L, 150 mg/L, and 200 mg/L of humic acid to investigate the role of humic acid concentration on the adsorption efficiency.

2.5 Adsorption of Humic Acid: Effect of PANI Dosage

An adsorption study was carried out using the same procedure as the one mentioned in section 2.4. This round, the humic acid concentration was fixed at 50 mg/L while the dosage of PANI varied from 2 g up to 12 g. Similarly, the spent PANI was separated using centrifugation and the supernatant (which consist of unadsorbed humic acid) was analysed using UV-visble spectrometry.

2.6 Fabrication of PANI Sheet

The spent PANI obtained after humic acid adsorption was deposited onto a filter paper/tissue paper/cotton cloth sheet *pre*-cut to 6.5 cm × 2.7 cm dimension. The deposition was done via the drop-casting method [34, 35]. Here, the spent PANI was casted drop-by-drop onto the substrate using laboratory dropper. Lastly, the PANI-deposited sheet was dried in an oven set at 80 °C for 30 mins. Photos of the blank sheets and those after being deposited with PANI particles are shown in Figure 1.
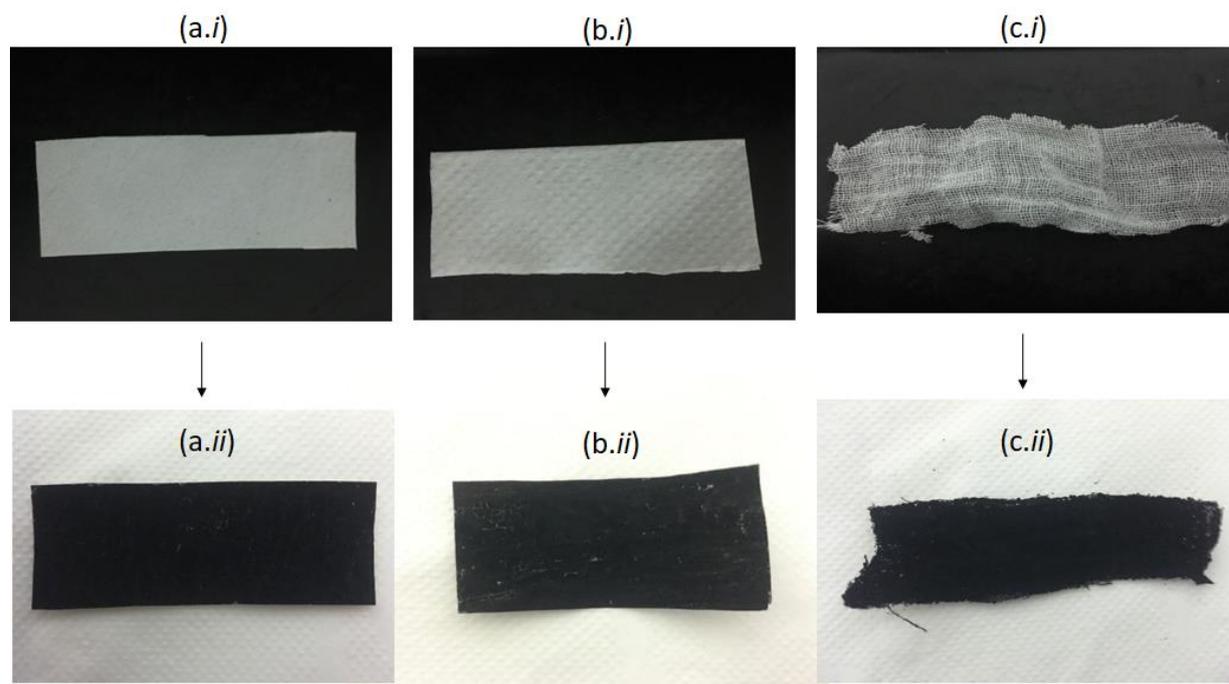
Figure 1. (a) filter paper, (b) tissue paper, (c) cotton cloth *pre*-cut to 6.5 cm x 2.7 cm dimension. (*i.* blank sheet, *ii.* After being deposited with PANI particles).

2.7 Determining Resistance and Electrical Conductance of the PANI Sheet

The resistance of the PANI-deposited sheet was determined using a digital multimeter [36, 37]. Crocodile clips were attached to the ends of a multimeter and connected to the PANI sheet. The resistance, R, of the PANI sheet was recorded in unit Ohms. The electrical conductance (in unit S) can then be calculated as the reciprocal of the resistance, $G = 1/R$. For comparison, the same steps were repeated for all types of PANI sheets. Figure 2 shows the standard setup used for measuring the resistance using a multimeter.
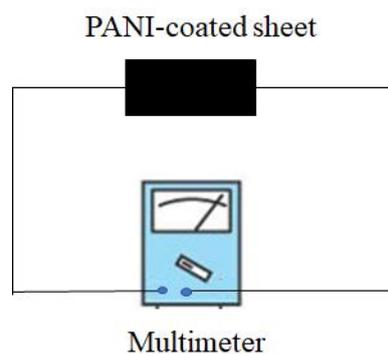

Figure 2. Schematic diagram showing the standard setup used to measure the resistance of the PANI-deposited sheet.

## 3. RESULTS AND DISCUSSION

3.1 Material Characterization

Figure 3 shows the transmission of electron microscopic (TEM) image of the synthesized PANI. The average particle size is ~352.96 nm. The PANI appears to be quite polydisperse and granule

in shape. The granule structure is the characteristic of PANI synthesized using strong acid [38]. Slight agglomeration was found in Figure 3; however, this might be a drying-artifact formed during the TEM sample preparation [39, 40].
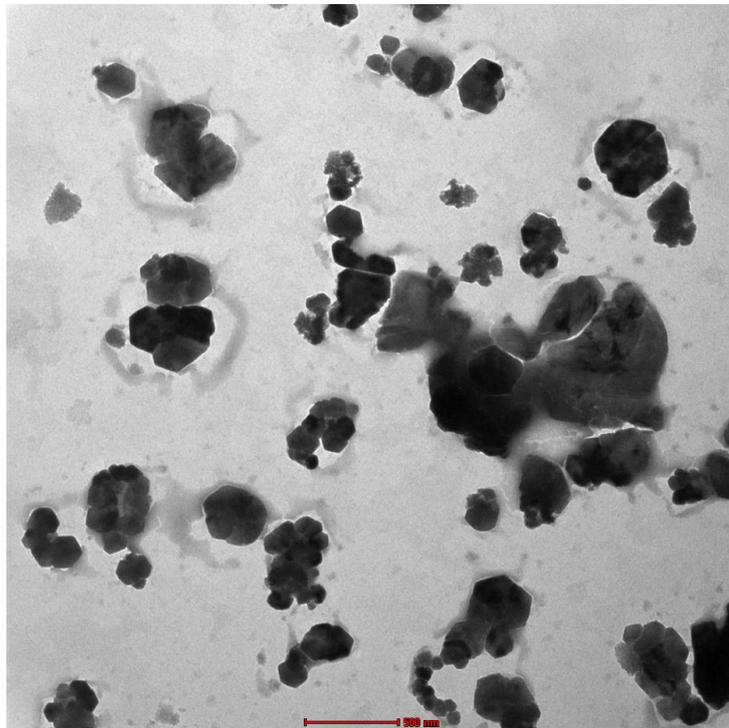

Figure 3. TEM image of the synthesized PANI.

FTIR spectra of the PANI (in the range of 500 to 4000 cm^{-1}) is shown in Figure 4. The broad peak around 3357 cm^{-1} was ascribed to the N-H stretching vibration [41]. The peak at 1639 cm^{-1} may ascribe to the C=C stretching mode [42-44]. Meanwhile, the peak at 1418 cm^{-1} and 1296 cm^{-1} were ascribed to the strong aromatic C-N stretching vibrations of the PANI [41, 45-47].
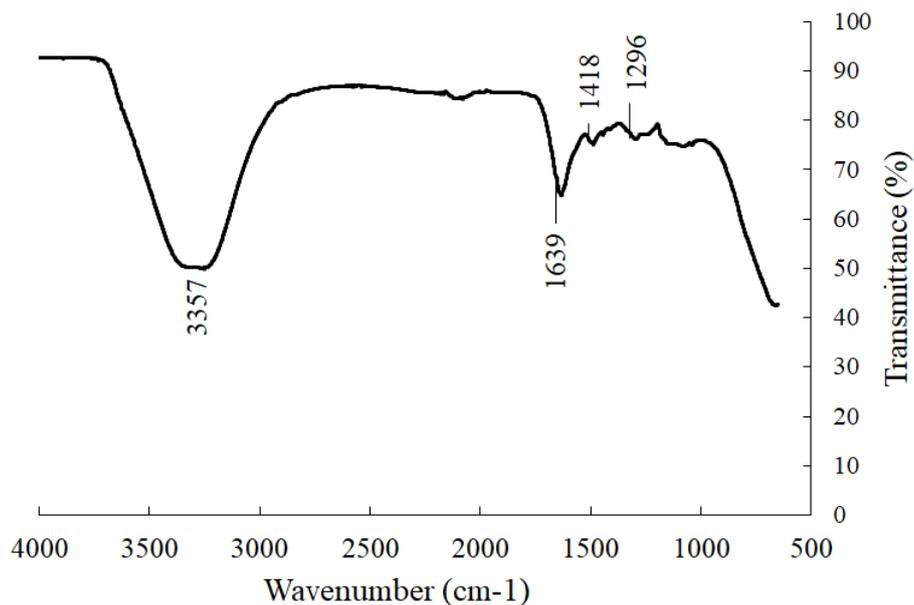
**Figure 4.** Fourier transform infrared (FTIR) spectra of PANI.

3.2   Humic Acid Removal by PANI

Next, the synthesized PANI was used for humic acid removal. The anionic humic acid is hypothesized to be favorably adsorbed onto the cationic PANI. As shown in Figure 5, the removal efficiency increased with the increase in initial humic acid concentration, whereby the highest removal efficiency (89.5 %) was achieved at 200 mg/L initial humic acid concentration; while 150 mg/L, 100 mg/L, and 50 mg/L of initial humic acid concentrations resulted in 85.9 %, 76.5 %, 49.3 % removal efficiencies, respectively. Despite being subjected to the same amount of adsorbent (*i.e.* 1.75 g of PANI), it is believed that the 200 mg/L of humic acid created a greater concentration gradient between the humic acid molecules in the bulk solution and the humic acid molecules on the PANI surface. This high concentration gradient serves as a driving force to promote the movement of humic acid from the bulk solution to the PANI surface, thus, inducing a better adsorption [48, 49]. Overall, this result has shown that PANI is a good adsorbent for humic acid.
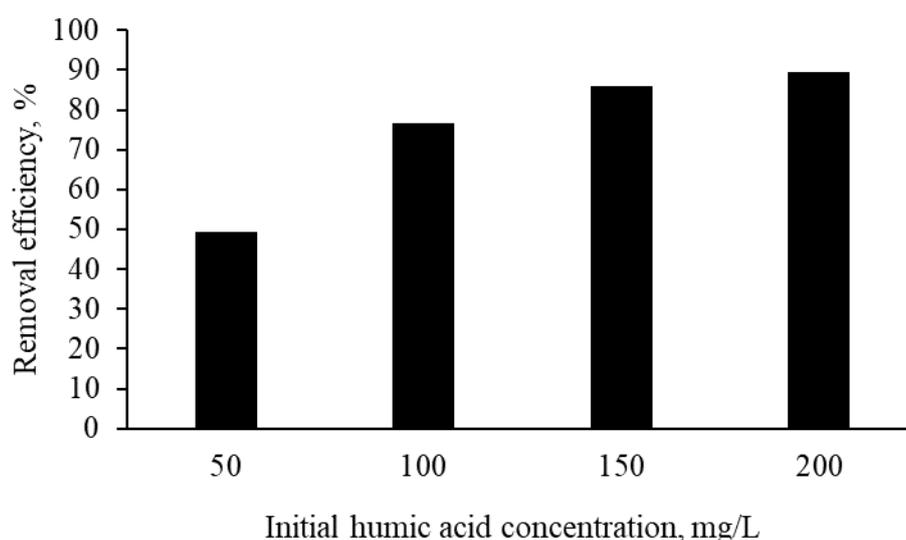
Figure 5. The removal efficiency at different initial humic acid concentrations
[Condition: 1.75g PANI]

Effect of PANI dosage on humic acid removal was studied by varying the PANI dosage from 2 g up to 12 g while maintaining the initial humic acid concentration at 50 mg/L. As shown in Figure 6, the humic acid removal efficiency improved as the PANI dosage was being increased. Basically, the removal efficiency has achieved an average of 9% increment for every time the dosage of PANI being increased by a magnitude of 2 g. This observation is true until a total of 8 g PANI being used. The removal efficiency surged up to ∼ 90% when the dosage of PANI was being increased to 8 g and stayed almost constant up to 12 g of PANI dosage. The increase in humic acid uptake with the increase in PANI dosage was attributed to the availability of more active-surface sites to absorb the humic acid [50]. However, there is no significant change in the removal efficiency from 8 g to 12 g of PANI. Such a phenomenon may ascribe to the conglomeration of adsorbent (PANI) particles which later, will lead to no increase in effective surface area [51].
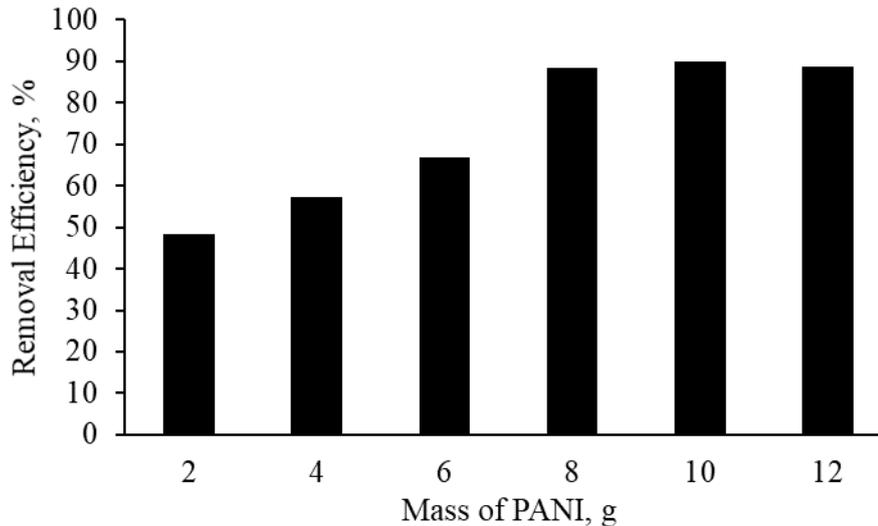
Figure 6. The removal efficiency at different initial PANI dosages [Condition: 50 mg/L humic acid]

3.3 Electrical Conductance of the Fresh and Spent PANI

Next, the electrical resistance, and thus electrical conductance of the freshly made PANI and spent PANI was evaluated by depositing the PANI onto three different sheets of substrates. Note that the spent PANI was the PANI that was already used for humic acid adsorption. Table 1 shows the obtained electrical resistance and conductance values. As expected, all the three sheets of substrates turned conductive upon being deposited with the freshly made PANI. Interesting to note that the fresh PANI-deposited filter paper exhibited a high electrical conductance (1.03×10^{-3} S), which is 2.43 times and 6.78 times greater than the electrical conductance of fresh PANI-deposited tissue paper and fresh PANI-deposited cotton cloth, respectively. Such observation may ascribe to the textural properties of the substrate material. As shown in Figure 1, the texture of cotton cloth appears to be sparser than the other two substrates; thus, its capability to capture/uphold the PANI is lesser. Also, both, tissue paper and cotton cloth exhibit less evenly distributed pore sizes which eventually lead to uneven PAN I deposition. On the other hand, the filter paper used here is a standard laboratory item which was manufactured with standard properties such as dimension, thickness and pore sizes, resulting in an even texture all over the paper. Hence, a more consistent PANI deposition can be a result

Table 1 Electrical resistance and electrical conductance of the fresh and spent PANI-deposited sheets

Sample	Electrical resistance, kΩ	Electrical Conductance, S
Filter paper (Blank)	∞	NA
Tissue paper (Blank)	∞	NA
Cotton cloth (Blank)	∞	NA
Fresh PANI-deposited filter paper	0.97	1.03×10^{-3}
Fresh PANI-deposited tissue paper	2.36	4.24×10^{-4}
Fresh PANI-deposited cotton cloth	6.60	1.52×10^{-4}
Spent PANI-deposited filter paper	14.40	6.94×10^{-5}
Spent PANI-deposited tissue paper	5.00	2.00×10^{-4}
Spent PANI-deposited cotton cloth	16.60	6.02×10^{-5}

Interestingly, it was found that the electrical conductance of each sheet dropped significantly after the PANI was spent for humic acid adsorption. For instance, the surface electrical conductance of PANI-deposited filter paper decreased immensely to 6.94×10^{-5} S from the initial

value of 1.03×10^{-3} S. This accounts to ~ 93.26% reduction in electrical conductance. Meanwhile, the PANI-deposited tissue paper and PANI-deposited cotton cloth also experienced a 52.83 % and 60.39 % decrease in electrical conductance, respectively.

One of the possible reasons for the conductance suppression is the involvement of the protonated part of PANI in the humic acid adsorption. In specific, the protonated imine and amine groups of PANI are favorably attracted to the dissociated humic acid molecules [52, 53]. It so happens that the conductivity of PANI strongly depends on the degree of protonation [41, 54, 55]. Hence, with more humic acid adsorbed onto the PANI, the remainder protonated sites on the spent PANI becomes lesser. This renders to poorer electrical conductance.

To further evaluate the effect of humic acid adsorption on the electrical conductance, the PANI particles used for different amounts of humic acid adsorption (as presented in Figure 5) were collected and deposited onto filter paper. Table 2 shows the corresponding electrical conductance of the spent PANI-deposited filter papers. Results show that the electrical conductance of PANI decreased substantially for humic acid adsorption. In particular, the extent of reduction increases with the initial humic acid concentration used for the adsorption study. Apparently, the more humic acid adsorbed onto the PANI particles, the lower the electrical conductivity of the spent PANI. Such observation further proved the detrimental effect induced by the adsorbed humic acid on the conductivity of PANI particles.

Table 2 Summary of electrical resistance and electrical conductance of spent PANI-deposited filter paper as compared to fresh PANI-deposited filter paper

Sample	Electrical resistance, kΩ	Electrical Conductance, S
Fresh PANI-deposited filter paper	0.97	1.03×10^{-3}
Spent PANI-deposited filter paper:		
50 mg/L humic acid	14.40	6.94×10^{-5}
100 mg/L humic acid	24.88	4.02×10^{-5}
150 mg/L humic acid	76.34	1.31×10^{-5}
200 mg/L humic acid	113.51	8.81×10^{-6}

To study the role of PANI dosage on the magnitude of electrical conductance, all the spent PANI presented in Figure 6 were deposited onto filter papers and measured for their electrical conductance. As shown in Figure 7, the electrical conductance of the spent PANI sheet was found to incline exponentially based on the dosage of spent PANI deposited onto the filter paper. Particular, 12 g of spent PANI-deposited filter paper exhibited 0.025 S electrical conductance, which is ~ 320 times higher than the 2 g spent PANI-deposited filter paper. Such observation is expected because the more PANI is deposited onto the filter paper indicates more active-PANI site available for electron flow. Thus, the filter paper deposited with greater dosage of PANI exhibited a higher electrical conductance.
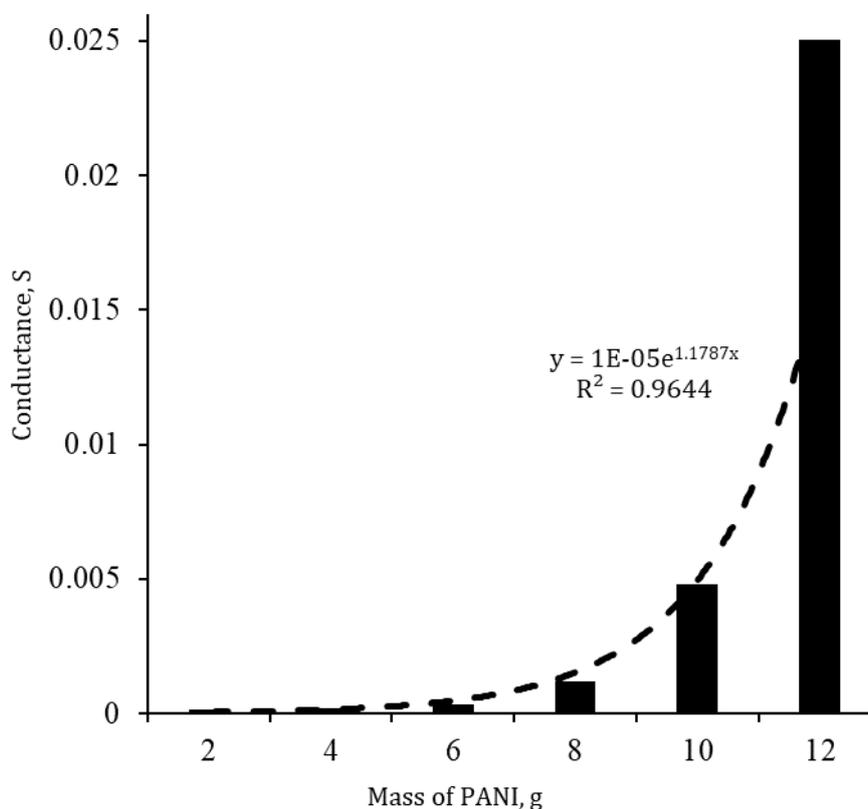
Figure 7. Electrical conductance of filter paper deposited with different amounts of spent PANI.

4. CONCLUSION

PANI of an average size of 352.96 nm was successfully synthesized via a simple oxidation method. The PANI was found to be effective in humic acid adsorption. In particular, the adsorption efficiency increased with the increase in initial concentration of humic acid due to the concentration gradient effect. Also, the adsorption efficiency was enhanced with the increase in PANI dosage. The spent PANI was not dumped towards the end of the adsorption application but reused as an electrical conductor. In this regard, a flexible electrical conductor was successfully fabricated by drop-casting the spent PANI onto a *pre*-cut filter paper sheet. The filter paper, which was originally not conductive, turned conductive upon being deposited with the spent PANI. Such a result suggests that the spent PANI can be reused for electrical application after being utilized for water remediation. Nevertheless, the electrical conductance of the spent PANI-deposited filter paper was found to be at least 14 times poorer than the fresh PANI-deposited filter paper. Also, the more the humic acid adsorbed onto the PANI, the lesser the magnitude of electrical conductance. This suggests that the adsorbed humic acid molecules have shielded the electron movement. Future work shall venture into a strategy to recover the electrical conductance of the spent PANI.

ACKNOWLEDGEMENTS

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