

Influence of Temperatures of Biochar Derived from Agro-waste onto Polyester Composite for improving the Water Effluent Discharge

Siti Samahani Suradi^{1}, Muhammad Fauzan Syafti Mohamed Fouzi¹, Zainatul Umairah Zainal Abidin¹, Dilaeleyana Abu Bakar Sidik¹, Izirwan Izhah², Nurul Izzati Mohd Ismail¹, Hazirah Bujang³, Nurmina Abdullah⁴*

¹Department of Science and Mathematics, Centre for Diploma Studies, Universiti Tun Hussein Onn Malaysia, Pagoh Higher Education Hub, 84600, Pagoh, Johor, Malaysia

² Department of Industrial Sciences and Technology, Universiti Malaysia Pahang Al-Sultan Abdullah, Lebuhr Persiaran Tun Khalil Yaakob, 26300 Kuantan, Pahang, Malaysia

³Department of Civil Engineering, Centre for Diploma Studies, Universiti Tun Hussein Onn Malaysia, Pagoh Higher Education Hub, 84600, Pagoh, Johor, Malaysia

⁴Department of Mechanical Engineering, Centre for Diploma Studies, Universiti Tun Hussein Onn Malaysia, Pagoh Higher Education Hub, 84600, Pagoh, Johor, Malaysia

Abstract. The biochar-based rice straw (RS) is the agro-waste from rice production which is one of the significant contributors to pollution. This study focused on altering RS biochar on polyester/polyvinylidene fluoride (RS-g-polyester) composites to improve the quality of water final effluent from industrial-based palm oil mills. Alteration of RS biochar-g-polyester composites was investigated at various RS calcination temperatures ranging from 300-900°C. The RS biochar-g-polyester composites were prepared using a dip-coating technique and characterized via spectroscopy (FTIR-ATR), Scanning Electron Microscopy (SEM-EDS), and tensile strength. Meanwhile, the performance of biochar RS-g-polyester composites was evaluated based on the pH, turbidity, and color removal of water effluent discharge. It was found that the biochar RS-g-polyester samples displayed different patterns in intensities and morphologies at different temperatures affecting the filtration of water effluent. Good interfacial compatibility of RS-g-polyester composite is also evidently seen via morphologies and mechanical properties. With consistent pH of waste effluent (8.83-8.89), the filtrate effluent discharge exhibits a significant reduction in color removal, 35% (~1150 ADMI), and turbidity, ~85% (0.9 8NTU) compared to initial water effluent discharge (~1800 ADMI). An increase in temperature has significantly improved the quality of water, thus it has potential use in the filtration of water treatment.

* Corresponding author: samahani@uthm.edu.my

1. Introduction

Malaysia is always proactive in promoting the effort of paddy farming for rice production to achieve its own-sufficiency food [1–3]. Since Malaysia still advocates almost 70% of local demand for rice productivity. As a result, grain crop-based paddy cultivation has generated massive amounts of rice straw annually, - as agro-waste, a residual by-product of rice production following harvest [3–5].

Rice straw (RS) is one of the solid agro-wastes that is a major contributor to the pollution of the environment involving air, water, and soil leading to health issues. This typical rice straw agro-waste management remains a challenge and a significant concern owing to conventional practices of stubble burning because of a lack of eco-friendly management, resulting in massive air pollution such as particulate matter, greenhouse gases, oxides nitrogen, sulfur, and volatile compounds. Hence, many researchers and engineers have focused on developing alternative applications-based rice straws that are sustainable for society [6–8].

Despite stubble burning, this solid agro-waste can be converted into cheap and eco-friendly products, like calcinated RS biochar residue, which was recently applied in the mitigation of wastewater treatment [9–11] and the remediation of organic and inorganic pollutants [4,10,12]. Biochar-based rice straw and stubbles are considered carbon-rich solid materials (Black -gold) from cheap sources of carbonaceous agro-waste, obtained when subjected to thermo-chemicals conversion at temperatures ranging from 300-900°C under limited oxygen conditions [2,4,13]. Biochar has been used in wastewater treatment, as well as an absorbent [14–16], membrane [17–20], and catalyst [18]. This is due to its environmental advantages such as a stable structure with more surface area, high exchange property, easier production, high carbon content, and eco-friendliness.

Numerous techniques are used for the modification of substrates to enhance their efficiency properties. Ghaffar et al., [17] successfully fabricated biochar/PVDF membranes to improve pollutant management in wastewater. Meanwhile, Chandra et al, [15] modified hydrogel/biochar as an absorbent for organic contaminant removal. It was found that there is limited study on the use of biochar-based RS onto polyester textile filters as a substrate for industrial effluent remediation. Therefore, this study focused on the synthesis and characteristics of the biochar-coated polyester/PVDF filter at different temperatures of calcination of biochar-based RS in terms of physiochemical and mechanical properties, as well as the effect of separation efficiency by using industrial final effluent discharged.

2. Material and Method

2.1 Materials

For the material of synthesis biochar, dry rice straw (RS) was used in this study and was collected from the community at a rice field at Sungai Balang, Muar, Johor Malaysia. Besides, polyester filter, 50 gsm was supplied by Capital Resources Engineering Sdn Bhd. Chemicals such as Natrium Hydroxide (NaOH), Polyvinylidene Fluoride (PVDF), Ethanol (C₂H₅OH), and Dimethylformamide (DMF) were supplied by R&M Chemicals, respectively.

2.2 Preparation of calcinated biochar at different temperatures and hydrolysis of polyester filter

Firstly, the rice straw (RS) was dried in the oven overnight at 60°C and then, it was calcinated at three different temperatures: 300 °C, 500 °C and 900 °C using the furnace Metrhom brand for 3 hours. After that, the RS biochar was crushed until fine and converted into powder. Next, the polyester filter samples were hydrolyzed using NaOH (1N) solution at 60°C for 2 hours. After that, the polyester filter samples were washed with distilled water for twices and soaked in the ethanol for 10 min before drying at 60°C for 24 hours.

2.3 Preparation of modified porous biochar derived from rice straw (RS) coated onto polyester

In the secondary stage, the PVDF, 0.3 g was poured into the DMF, 10 ml, and stirred for 30 minutes. Then, the RS biochars, 0.5 g at different temperatures were added to the mixture of polymerized PVDF/DMF solution, and then, the solution was stirred well dispersion between biochar and polymerized PVDF/DMF. Next, the solution was poured into the glass petri dish. Finally, the polyester filter samples were immersed in the mixture solution and were dried at 60 °C for 7 hours to form the biochar rice straw grafted polyester (RS-g-polyester) composites.

2.4 Characterisation of modified rice straw coated onto polyester

The samples were analyzed in terms of their surface chemistry and morphology, which were conducted through Attenuated Total Reflectance of Fourier transform infrared (ATR-FTIR), and Scanning Electron Microscope - Energy Dispersive Spectroscopy (SEM-EDS), Agilent model, operated at 20kV and high magnification and ATR-FTIR ranging from 700-4000 cm^{-1} , respectively. Meanwhile, the mechanical tests including tensile strength and elongation were analyzed via a universal testing machine. The performance of water effluent discharge was also quantified in terms of pH, decolorization effect, and turbidity tests. The pH values were determined by using a pH meter. Meanwhile, the decolorization effect and turbidity tests were evaluated via a water tester, DR6000 Ultraviolet-visible (UV-VIS) spectrophotometer, and turbidity tester (model TL2300). The decolorization was referred to the standard program, color unit American Dye Manufacturers Institute (ADMI), 10 mm.

3. Result and Discussion

3.1 Surface chemistry of unmodified and modified biochar rice straw coated with polyester at different temperatures

The FTIR spectrums between untreated polyester, treated polyester, and biochar RS-g-Polyester at 300°C, 500°C, and 900°C were used to identify different functional groups on its surface as shown in Fig. 1. In the given spectra, the band up to 3335 cm^{-1} is attributed to the presence of hydroxyl groups, (-OH). A slight difference in peaks can be seen at the band within 1680-1750 cm^{-1} indicating the presence of carboxyl and ketones stretching, while C=C stretching, displays the presence of aromatic hydrocarbons. A significant difference between polyester filter composite before and after modification with RS biochar was presented in the band range of 1450-1100 cm^{-1} attributed to C-N, C-O, and C-O-C functional groups [9,11]. It was found that the intensity of peaks in the band range of 900-

700 cm⁻¹ associated with the aromatic hydrocarbons and silica (SiO₂) is increased with an increase in the calcination temperature of RS [21]. This is due to the reduction of hydroxyl (-OH), carboxylic, and aliphatic (-CHn) stretching, possibly the amount of SiO₂ in biochar RS rises with the increase of temperature up to 900°C [22].

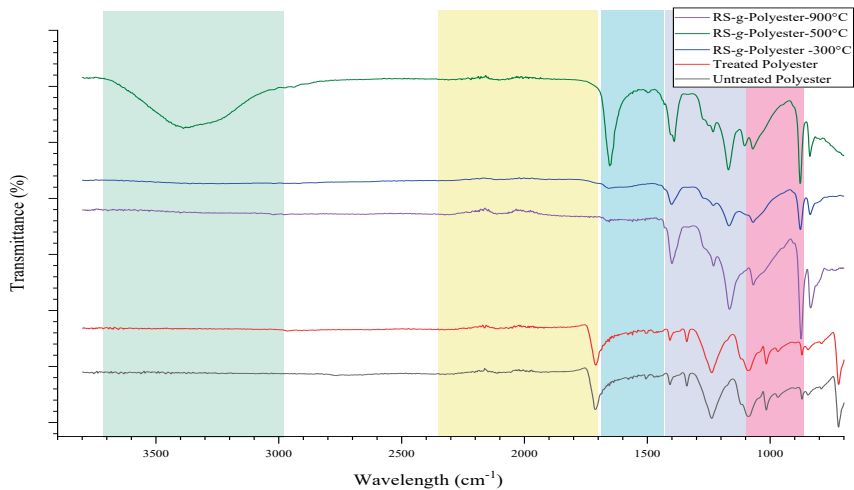


Fig. 1. Surface Chemistry of untreated Polyester, Treated Polyester, and biochar RS-g-Polyester at Different Temperatures

3.2 Morphologies of unmodified and modified Biochar Rice straw coated with Polyester at different temperatures

Fig. 2(i)(a) illustrates a detailed morphological characteristic of the RS biochar at different temperatures. It can be seen from Fig. 2(i)(a-c), that the change of surface morphology of the RS biochar concerning the calcination temperatures was raised. The pores in the materials became prominent. As the temperature rose to 300°C, numerous small and dense fragments aligned with the fissures and macropores started to appear on the surface of RS biochar. Further increase in temperature up to 500 °C, the fissures and macropores became enlarged, well-developed pores and size with the appearance of micropores. Further increment in temperature up to 900°C, the size of macropores was decreased and dense due to the deposition of condensed aromatic structures [21].

Meanwhile, Fig. 2(ii)(e-g) shows the deposited of RS biochar onto the Polyester with the presence of PVDF. Further imaging of SEM as shown in Fig. 2 (ii)(f-g), unveils the presence of agglomerated particles of RS biochar indicating the successful deposition of biochar on the polyester/PVDF substrate, as evidenced of the facile synthesis via dip-coating method [10].

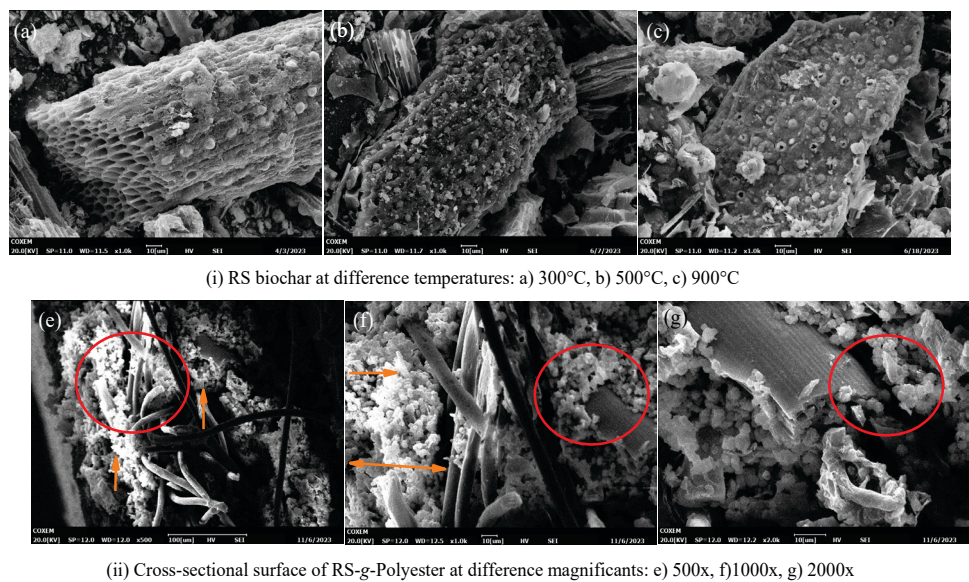


Fig. 2. Surface Morphologies:i) Top-view of RS biochar at different temperatures ranging from (a)300;(b)500; (c)900 at 1000x magnificant, ii)Cross-section view of RS-g-Polyester at difference magnificant: (e)500x; (f)1000x; (g) 2000x

3.3 The Biochar RS-g-Polyester Mechanical Properties

The mechanical properties of all biochar RS-g-Polyester composite filters were evaluated in terms of tensile strength and elongation as shown in Table 1. As expected, the tensile strength of the modified polyester filters with additional RS biochar decreased as the RS calcination temperature increased [23]. The treated polyester exhibits low tensile and elongation due to the enhancement of hydroxyl groups after hydrolysis leading to a significant loss of strength and interfacial compatibility compared to untreated polyester.

The biochar RS-g-polyester slightly increased at 900°C, most probably due to homogenous and fine particle size has caused biochar easily to disperse and aggregate, decreasing interfacial compatibility between biochar and polyester composite, resulting in uneven stress distribution, thus reducing the mechanical properties. These biochar RS-g-Polyester still have good stability and stretchability properties [24].

Table 1. The effect of mechanical tensile strength, elongation, and pH of the RS-g-Polyester after filtration by using water effluent discharge from palm oil mill industry

Parameter	Untreated Polyester	Treated Polyester	RS-g-Polyester (300 °C)	RS-g-Polyester (500 °C)	RS-g-Polyester (900 °C)
Tensile Strength	4.79± 0.155	2.22±0.023	2.563±0.468	2.18±1.170	2.67± 0.290
Elongation	18.15± 2.255	9.76±0.092	3.938±0.651	4.035±0.347	4.042± 0.222
pH	8.83±0.023	8.89±0.018	8..93±0.013	8..86±0.020	8..92±0.010

3.4 Effect of calcination temperatures on the efficiency of pH, color removal, and turbidity

Fig. 3 (a-b) presents the effectiveness of color removal and turbidity of the RS-g-polyester composites, which vary in temperature at 300 °C, 500°C, and 900 °C. The obtained result as seen in Fig. 3(a) shows a gradual increase in color removal and turbidity efficiency with increasing temperature of calcination of RS. It was found that the RS biochar coated onto polyester was capable of reducing the color with the efficiency reaching ~35% with the value of color by ~1511 ADMI, compared to the initial value of water effluent discharge (~1798 ADMI) as seen in Fig. 3(b). Simultaneously, the turbidity effectively affects the quality of water effluent discharged, which efficiency reached ~85% at 900°C, while the turbidity value significantly dropped from 18 NTU to 0.98 NTU (Fig. 3(b)).

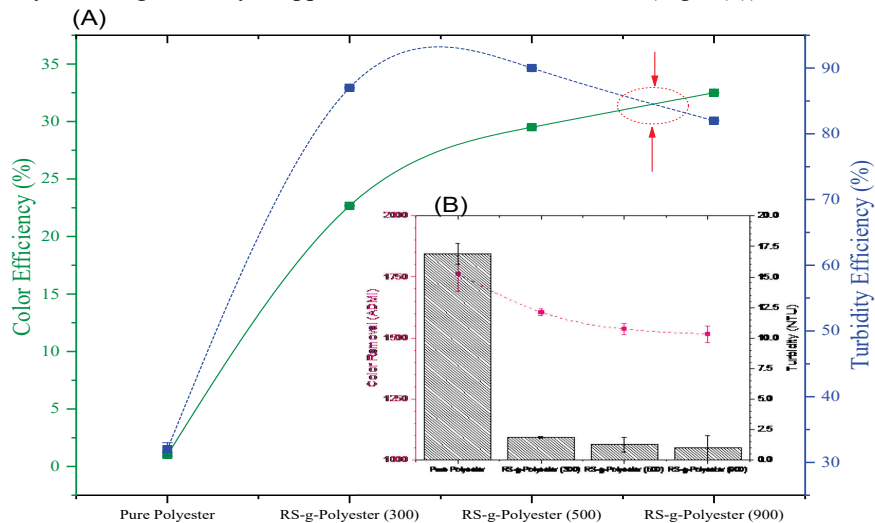


Fig. 3. The effect of color removal and turbidity on the filtration of RS-g-Polyester at different temperatures by using water effluent discharge from palm oil mill industry;(A) The efficiency of color removal and turbidity;(B) Amount of color removal and turbidity after filtration

This suggests that an increase in temperature, led to higher surface area and greater accessibility of RS biochar, resulting in strong negative RS-g-polyester surface charges. Since the source of the performance test used water effluent from a palm oil mill, it was also found that the filtrate effluents remained consistent at pH 8.83-8.92 as shown in Table 1 suggesting the presence of organic compounds and color pigments such as tannin, lignin, and carotenoid [25]. Thus, the adjustment of RS-g-polyester to be more surface negatively charged is one of the ideal approaches to improve the water effluent discharge

4. Conclusion

Additional RS biochar onto polyester composite has shown promising feasibility in improving the quality of water effluent discharged. Manipulating the calcination temperature of RS exhibited a significant difference in the intensity of peaks and morphologies. Besides, the morphologies and mechanical properties show good interfacial compatibility between RS biochar and polyester composite. The findings show that the effect of temperature has improved the surface charge which resulted in better performances, with the enhancement of efficiency of color removal and turbidity up to ~35% and 85%, respectively.

This research was supported by Universiti Tun Hussein Onn Malaysia (UTHM) through Tier 1(vot Q128). The authors would also like to thank Universiti Tun Hussein Onn Malaysia for its support.

References

1. B. Q. Lap, N. V. D. Thinh, N. T. Q. Hung, N. H. Nam, H. T. T. Dang, H. T. Ba, N. M. Ky, H. N. A. Tuan, *Water. Air. Soil Pollut.* **232**, (2021).
2. S. Y. Foong, Y. H. Chan, B. L. F. Chin, S. S. M. Lock, C. Y. Yee, C. L. Yiin, W. Peng, S. S. Lam, *Bioresour. Technol.* (2022).
3. Y. Singh, S. Sharma, U. Kumar, P. Sihag, P. Balyan, K. P. Singh, O. P. Dhankher, *Sci. Total Environ.* (2024).
4. R. Kumar, P. Sharma, P. K. Sharma, P. K. Rose, R. K. Singh, N. Kumar, P. K. Sahoo, J. P. Maity, A. Ghosh, M. Kumar, P. Bhattacharya, A. Pandey, *J. Environ. Manage.* **343**, (2023).
5. R. B. R. Firdaus, M. Leong Tan, S. R. Rahmat, M. Senevi Gunaratne, *Cogent Soc. Sci.* (2020).
6. G. Y. Rani, T. Jaya Krishna, *Mater. Today Proc.* **60**, (2022).
7. S. Kirubai, S. Padmavathy, N. Ganesh, K. Rajaguru, *Mater. Today Proc.* (2022).
8. A. Bhatnagar, A. Singhal, H. Tolvanen, K. Valtonen, T. Joronen, J. Konttinen, *Waste Manag.* **138**, 298 (2022).
9. H. R. Robles-Jimarez, L. Sanjuan-Navarro, N. Jornet-Martínez, C. T. Primaz, R. Teruel-Juanes, C. Molins-Legua, A. Ribes-Greus, P. Campíns-Falcó, *Sci. Total Environ.* **805**, (2022).
10. P. Bhadoria, M. Shrivastava, A. Khandelwal, R. Das, S. Langyan, B. Rohatgi, R. Singh, *Sustain. Chem. Pharm.* **29**, (2022).
11. Y. Lim, B. Kim, J. Jang, D. S. Lee, *J. Environ. Chem. Eng.* **11**, (2023).
12. S. A. Hassan, A. S. Darwish, H. M. Gobara, N. E. A. Abed-elsatar, S. R. Fouda, *J. Mol. Liq.* (2017).
13. Shemawar, A. Mahmood, S. Hussain, F. Mahmood, M. Iqbal, M. Shahid, M. Ibrahim, M. A. Ali, T. Shahzad, *Sci. Rep.* **11**, (2021).
14. N. Karakoyun, S. Kubilay, N. Aktas, O. Turhan, M. Kasimoglu, S. Yilmaz, N. Sahiner, *Desalination* **280**, 319 (2011).
15. S. Chandra, I. Medha, J. Bhattacharya, *Sci. Total Environ.* **712**, (2020).
16. J. Lang, L. Matějová, A. K. Cuentas-Gallegos, D. R. Lobato-Peralta, K. Ainassaari, M. M. Gómez, J. L. Solís, D. Mondal, R. L. Keiski, G. J. F. Cruz, *J. Environ. Chem. Eng.* **9**, (2021).
17. A. Ghaffar, X. Zhu, B. Chen, *Environ. Pollut.* **233**, 1013 (2018).
18. Z. Zhou, L. Xu, X. Zhu, Q. Wang, X. Meng, T. Huhe, *Chemosphere* **337**, (2023).
19. O. Kazak, G. K. Akkaya, A. Tor, *J. Environ. Chem. Eng.* **11**, (2023).
20. M. Naidu Subramaniam, S. Zhou, G. Zhang, J. C. Manayil, Z. Wu, *Sep. Purif. Technol.* **352**, 128 (2025).
21. S. Chandra, J. Bhattacharya, *J. Clean. Prod.* **215**, 1123 (2019).
22. A. S. El-Hassanin, M. R. Samak, S. R. Radwan, G. A. El-Chaghaby, *Environ. Nat. Resour. J.* **18**, 283 (2020).

23. D. She, J. Dong, J. Zhang, L. Liu, Q. Sun, Z. Geng, P. Peng, *Compos. Sci. Technol.* (2019).
24. M. Yang, H. Xu, X. Hou, J. Zhang, Y. Yang, *J. Environ. Manage.* **188**, 26 (2017).
25. Y. H. Tan, P. S. Goh, A. F. Ismail, B. C. Ng, G. S. Lai, *Chem. Eng. J.* **308**, 359 (2017).