Available online at
https://journal.uitm.edu.my/ojs/index.php/MJCET

e-ISSN: 2682-8588

Malaysian Journal of Chemical Engineering & Technology 8(2) 2025, 158–167

Malaysian Journal of Chemical Engineering and Technology

Formulation of polymer composite from starch/PVA and crosslinking with silica from rice husk ash for polymer flooding

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ARTICLE INFO

Article history:
Received 17 August 2025
Revised 21 August 2025
Accepted 27 October 2025
Online first
Published 31 October 2025

Keywords:
Polymer composite
Starch
Silica
Rice husk ash
Polymer flooding
Enhanced oil recovery

DOI:
10.24191/mjcet.v8i2.8543

ABSTRACT

This research project focuses on the creation of a polymer composite by combining potato starch, polyvinyl alcohol (PVA), and silica from rice husk ash (RHA) for polymer flooding in enhanced oil recovery (EOR). This study aims to overcome the limitations of the polymer use for EOR, such as polymer degradation, shear-thinning behaviour, and viscosity control of the polymer. The polymer blend of starch/PVA was crosslinked with silica from RHA, which is expected to enhance the shear-thinning behaviour and control the viscosity of polymer solutions. The investigation involves varying silica concentration to observe its impact on shear rate and viscosity. The methodology includes the extraction of silica from rice husk and the preparation of the polymer composite solution, followed by characterisation of the polymer using Fourier Transform Infrared Analysis and viscosity analysis. The polymer composite was expected to withstand the harsh conditions in the reservoir, which can help to enhance the oil recovery. The polymer composites showed stability in viscosity as the temperature rose, indicating high thermal stability, with no effects from the salinity in 2.5% and 5% saline concentration. In conclusion, the presence of silica, particularly 3%, can improve the rheological properties of biopolymer solutions, making them more stable under reservoir conditions such as high salinity and temperature.

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https://doi.org/10.24191/mjcet.v8i2.8543

1. INTRODUCTION

Chemical flooding enhanced oil recovery (EOR) is considered highly advantageous due to its higher efficiency and lower capital cost. EOR chemicals, such as alkali and surfactants, can reduce interfacial tension in the reservoir by modifying the rock-fluid contact. They can also alter the wettability of the oil-rock-brine contact (Abbas et al., 2020). Polymer flooding, on the other hand, is extensively used to increase the viscosity of the injectant and, as a result, the mobility of the reservoir fluids. As a result, viscous fingering is reduced or eliminated, conformity control is improved, and sweep efficiency is increased. According to field studies, polymer flooding can recover an additional 5 – 30% Original Oil in Place (OOIP) (Saboorian-Jooybari et al., 2016). Studies conducted in the field of EOR have demonstrated that adding starch to waterflood sites results in a 6 – 8% increase in cumulative oil production, compared to synthetic polymer (commercial polyacrylamide) flooding (Singh et al., 2017). Potato starch forms a paste having high viscosity that endears it as a possible agent for EOR since the mobility ratio of the displacement process is reduced. Other properties are its binding capacity and the formation of gel within 50 – 80 °C. The gelatinisation temperature can be modified to ensure its use within the confines of petroleum reservoir temperatures. Potato starch contains mainly amylose and amylopectin, with minor components including protein and lipids. Because of their availability, low cost, and benign nature, bio-based materials have recently sparked attention in a variety of disciplines (Piana et al., 2020). This is because the effectiveness of native starch is negatively affected by harsh conditions such as high pressure, temperature, and salt in a hydrocarbon reservoir. In contrast to native starch, crosslinked starch produced by grafting vinyl monomers onto starch displays reduced microbial attack, enhanced rheological behaviour of the solution, and rising thermal stability (Leslie et al., 2005). Furthermore, the addition of silica particles reinforces the polymer and boosts its resistance to salt. This improved composite is effective for biopolymer flooding operations in tough reservoir conditions (Abbas et al., 2020).

Natural polymer solutions show shear-thinning performance, where the viscosity increases with increasing shear rate, as has been noticed when using other high molecular weight polymers (Mudgil et al., 2014). When the polymer chain is stretched in distilled water due to the force of repulsion between the negative charges in the salt chain, there is a reduction in the polymer viscosity (Musa et al., 2021). The viscosity of the polymer is directly proportional to the increase in polymer concentration (Samanta et al., 2011). The viscosity of polymer solutions is highly sensitive to salinity changes. High salinity can lead to a reduction in the viscosity of the polymer solution. This is critical in EOR because maintaining high viscosity is essential for improving sweep efficiency and controlling the mobility of the injected fluid. Increasing the concentration of natural polymers generally enhances the viscosity of the solution. However, there is an optimal concentration beyond which further increases may lead to diminishing returns or issues like excessive pressure drops. Reservoirs can have varying salinity levels, and it is essential to choose a polymer that remains effective under the specific salinity conditions of the reservoir. Compatibility with the reservoir's brine is crucial for maintaining the desired rheological properties. The concentration of natural polymers needs to be optimised based on the specific reservoir conditions. Too high or too low concentrations may result in inadequate mobility control and inefficient oil displacement (Samantha et al., 2011).

https://doi.org/10.24191/mjcet.v8i2.8543

As the temperature increases, the average speed of the molecules in the liquid increases, and the time they spend in contact with their nearest molecules decreases (Khan et al., 2019). Al Soubaihi et al. (2025) added different amounts of silica to a methacrylate polymer and studied the effect of silica content on its thermal properties. As a result, as the silica content increased, the composite's decomposition temperature also increased, while the slope of the corresponding curve decreased during the decomposition process, implying that the inclusion of silica in composites reduced decomposition rate while improving heat resistance and thermal stability. This is due to silica's strong heat stability and chemical bonding, which form a crosslinking network with the polymer matrix. When heated, the crosslinking network structure prevents the polymer molecular chain from moving. The polymer is utilised to increase the viscosity of the mobile aqueous phase. The efficacy and success of the polymer immersion process can be measured by the polymer's ability to maintain viscosity while spreading in the reservoir (Reddy et al., 2011). Polymers, in particular, natural polymers, are susceptible to degradation. The viscosity reduction is due to uncoiling and alignment of natural polymer chains when exposed to shear flow (Khan et al., 2019). In their research, Ji et al. (2018) used the sol-gel process to create silica nanocomposites, and they discovered that the tensile strength, elongation at break, impact strength, and flexural strength of silica nanocomposites rose as the silica concentration increased. Ji et al. (2018) also investigated how the size of silica particles affected the mechanical properties of the nanocomposites they created. As the particle size of the loaded silica rose, the storage modulus dropped with increasing strain. This is because smaller particles have a bigger filling-polymer interface area, resulting in chemical connections between the filler and polymer at the interface. As the interface area rises, more chemical bonds are created, and the tensile strength increases. As a result, silica nanoparticles derived from sustainable materials are being researched to replace the present ones (Agi et al., 2018). Thus, this study pushes the boundaries of research by synthesising silica nanoparticles from rice husk ash (RHA) through the sol-gel method and then testing them for EOR applications. In this study, silica nanoparticles were synthesised from RHA, then mixed with polyvinyl alcohol and potato starch to create a polymer composite. The objectives are to control the viscosity of the polymer solution and optimise it for efficient displacement of oil within the reservoir. The stability of the polymer molecules in the injected solution was also enhanced, ensuring that the desired rheological properties are maintained throughout the injection process.

2. METHODOLOGY

2.1 Materials

Corn starch was purchased from Chemiz (M) Sdn. Bhd. Polyvinyl alcohol (PVA) with hydrolysis degree of 87 – 89 % (MW: 85000 – 124000), tetraethyl orthosilicate (TEOS) with 99 % purity and the potato starch were all purchased from Sigma-Aldrich (M) Sdn. Bhd, sodium hydroxide pellets (NaOH) were purchased from R&M Chemicals, Subang. BT Science Sdn. Bhd. supplied cethyltrimethylammonium bromide (CTAB), hydrochloric acid (HCl) with 37 % purity and ethanol. Laboratory-grade sodium chloride (NaCl) from R&M Chemical, Subang, was used to prepare the brine. RHA was obtained from Kilang Beras Bagan Serai Sdn. Bhd., Malaysia.

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2.2 Methods

Extraction of silica from RHA

An amount of 25 g of raw RHA was added to 250 ml of distilled water. Then 8 g HCl was added to the mixture to remove metal impurities. This step is called the pre-cleaning process. The solution was heated and stirred at 90 °C with 650 rpm for 1 h. The mixture was left to cool overnight at room temperature, and it was then filtered using filter paper. The next process is called the leaching process, where the filtrate was mixed with 250 ml (1M NaOH), heated at 80 °C, and stirred at 600 rpm for 1 h. The mixture was left overnight to cool at room temperature. The solution was filtered using filter paper. Brownish liquid discharged at pH 14 was obtained from the filtration process. Then 1N HCl was prepared for the gelling process. The brownish liquid was titrated with 1N HCl until it solidified into a gel texture and reached pH 7. The gel was left to age at room temperature for 24 h. After that, the gel was stirred vigorously using a magnetic stirrer at 1500 rpm to break into smaller pieces and washed using 400 ml of distilled water. The washing process was repeated 4 times until a clear (whitish) gel was obtained. The washing process was conducted to remove NaCl from the gel to obtain high purity of silica and water. The gel was then dried in the oven. The process flow diagram is shown in Fig. 1.

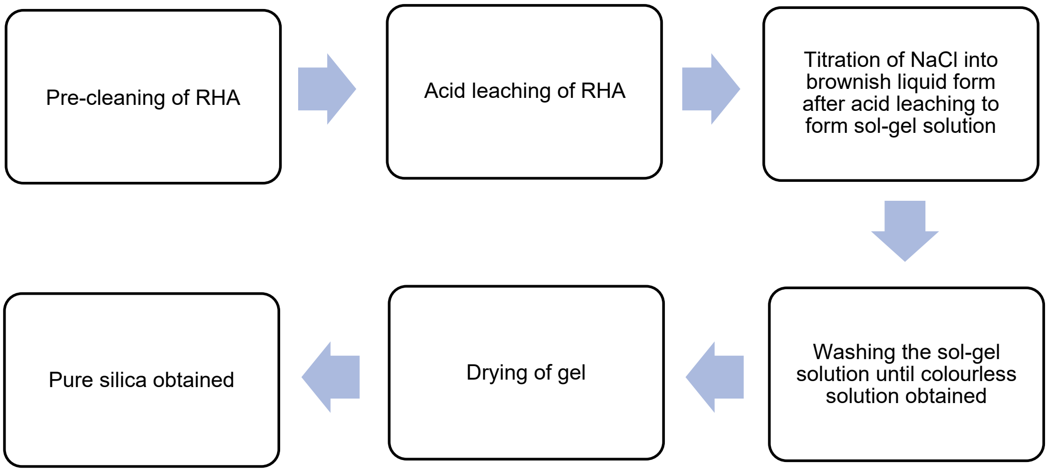

Fig. 1. Process flow diagram of the silica extraction from rice husk ash (RHA)

Source: Author's own illustration

Preparation of PVA and starch solution

For the preparation of the PVA solution, 10 g of PVA powder was mixed with 90 g of distilled water at 90°C for 4 h with continuous stirring at 400 rpm until it became a homogenous solution. It was then cooled down to room temperature. To prepare 5 % starch solution, 5 g potato starch was mixed with 100 ml of distilled water at a temperature of 60 °C and 350 rpm for 1 h.

Preparation of different saline concentrations

For the preparation of a 2.5 % saline concentration, 6.25 g NaCl was dissolved in 250 ml distilled water to achieve 25000 ppm of NaCl solution. For the preparation of a 5.0% saline concentration, 12.5 g NaCl was dissolved in 250 ml distilled water to achieve 50000 ppm of NaCl solution. Both solutions were dissolved by continuous stirring.

Preparation of polymer solutions

Both prepared solutions of PVA and starch were combined and stirred continuously at 400 rpm with heating at 60 °C for 7 h. Silica gel was added at 0 – 0.1 wt.% of the total mixture. Exactly 10 ml of HCl was added as a catalyst for the sol-gel method. Then, the solution was cooled down to room temperature. Potato Starch Polymer Nanocomposite (PSPNP) were produced with this formulation; 5 wt.% Potato Starch (PSP) and 0 wt.% silica, 5 wt.% PSP and 1 wt.% silica, 5 wt.% PSP and 5 wt.% silica. Also, the solutions for PSPNP were produced at two saline concentrations of 2.5 wt.% and 5.0 wt.%. The rheology of the three polymers was determined using thermal analysis and rheology instrument, viscometer (Model 35SA) at shear rate ranges of $0.1 - 500\ s^{-1}$, and the viscosity measurement was conducted at 50 and 100 °C (Orodu et al., 2019). The viscosity was then calculated using Eq. (1).

$$nN = S \times \theta \times f \times C \tag{1}$$

where: nN is Newtonian viscosity, S is speed factor, θ is dial reading, f is spring factor, and C is rotor-bob factor.

Characterisation of polymer – FTIR analysis

Fourier Transform Infrared Spectroscopy (FTIR) (Perkin–Elmer Spectrum 2000) was used to analyse the functional groups of polymers. The analysis was done at a wavelength between 400 cm^{-1} and 4000 cm^{-1}.

3. RESULTS AND DISCUSSION

3.1 FTIR analysis

FTIR spectra for the polymer composites with various concentrations of silica is shown in Fig. 2. There are several peaks discovered for all polymers, where they show the fingerprints of the polymer's material. In the case of hydrophilic silica sol-gel, the absorption peak O-H observed at around 3400 cm^{-1} is assigned to free or adsorbed water, the band at around 1083 cm^{-1} corresponds to the asymmetric stretching vibration of the Si-O-Si (siloxane) bond, and the band at around 1650 cm^{-1} refers to the presence of the vibrational mode of the Si-OH (silanol) bond (Wagh et al., 2015). The C-O stretching peak of the PVA backbone arises at roughly 1200 cm^{-1} (Hari Gopi et al., 2019). Based on the figure, as the silica concentration increases, the siloxane bond for each of the polymers stretches towards the peak of the siloxane bond of silica. The closer the stretch towards the peak of siloxane of silica indicates the higher the concentration of silica in the polymers. The silica-specific bands become more pronounced, and interactions between silica and the polymer matrix can lead to shifts and changes in the polymer's characteristic absorption bands. With the increase in silica concentration, the intensity of O-H and Si-OH bonds increases.

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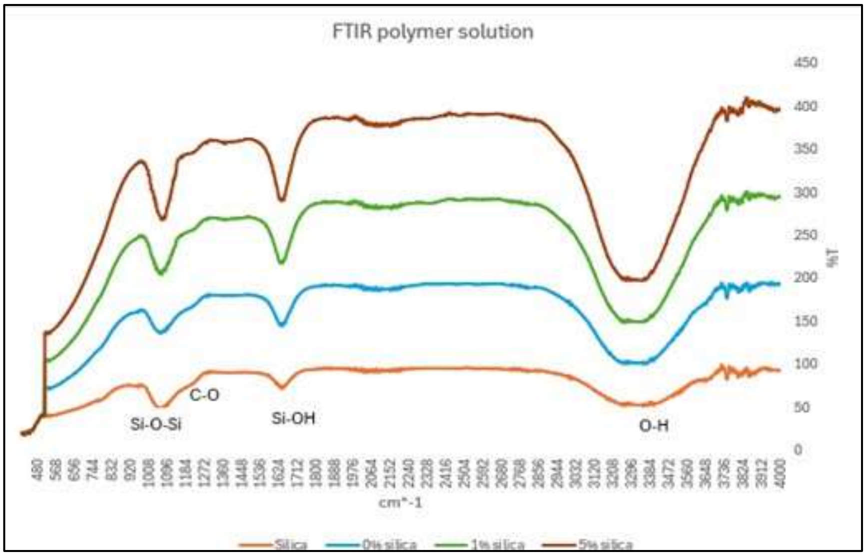

Fig. 2. FTIR spectra for polymer composite with various concentrations of silica

Source: Author's own data

3.2 Effect of salinity and temperature on the rheology of biopolymer nanocomposites

The relationship between viscosity and shear rate at 50 °C and 100 °C at various silica concentrations is shown in Figs. 3, 4, and 5. Based on Fig. 3, there was no significant effect of salinity on the viscosity of the biopolymer, as at the lower shear rate, the polymer composites in 5 % salinity had higher viscosity compared to those in 2.5 % salinity. However, the composite exhibited shear-thinning behaviour for all salinities. This is due to the biopolymers' non-ionic nature and their tolerance of various molecules. This non-ionic nature tends to improve the viscosity of the biopolymer regardless of the salinities. On the aspect of temperature, there was no decline in viscosity at 100 °C compared to 50 °C at low shear rate. Furthermore, the increase in the shear rate from 170/s resulted in a drastic drop in the viscosity of the polymer composite regardless of the temperature and salinity. The heat-resistant property of PSPNP is possibly attributed to its crosslinking polymer structure from the effect of the silica. Based on similar findings, the viscosity tends to decrease as the shear rate increases, providing better mobility control and improving the displacement of oil. This helps to prevent fingering and ensures a more uniform sweep of the reservoir (Orodu et al., 2019).

The range of viscosity for all the polymer composites at various concentrations of silica was found to be similar to that obtained by Jiang et al. (2016). The viscosity trend at 50 °C for salt concentrations of 2.5 % and 5 % is also consistent with Jiang et al. (2016). Based on Figs. 3, 4, and 5, there was no decrease on the viscosity with the increase in salinity at 5 % NaCl compared to 2 %, which shows that the presence of silica has reduced the shielding effect of sodium ions where the nanocomposites' interactions remain undisturbed. This phenomenon is ascertained by Shi et al. (2012) and corroborated by the electric double

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layer and the decrease in Debye's length of the nanocomposite. However, this trend was reversed at 100 °C. Again, the action of NaCl at 50 °C was adequately described by Herrera et al. 2017, where he explained the inverse link between viscosity and temperature to the transition from particulate to molecular networks (gel networks). As temperature increases, hydrodynamic diameter, storage modulus, loss modulus, and complex viscosity decrease (Orodu et al., 2019). However, results show that a similar viscosity profile at 100 °C with 50 °C may be due to the stronger inter- and intra-molecular bonds of the nanocomposite, and an increase of starch-water hydrogen bond (Shi et al., 2012). In addition, cations affect the starch structure and inhibit gelatinisation temperature (Oosten, 1982). Thus, the incorporation of silica nanoparticles helps to maintain the viscosity of PSPNP. The shear-thinning behaviour of polymers means that as they are injected through narrow pores and high-shear regions of the reservoir, their viscosity must remain higher, making it easier to inject the polymer solution into the reservoir. A higher viscosity polymer solution, which is the target, reduces the mobility of the displacing fluid (water with polymer) relative to the displaced fluid (oil). This improved mobility ratio helps in sweeping the oil more efficiently and reduces the chances of the displacing fluid bypassing the oil, leading to more uniform displacement. Due to higher temperatures in the reservoir, it can reduce the viscosity of the polymer solution. Based on the obtained result, adding silica to the polymer solution had increased the thermal and mechanical stability of biopolymer solutions, allowing them to withstand harsh reservoir conditions better than biopolymer solutions alone (Pope, 2011). This has been proven by the viscosity results at various silica concentrations, where the increase in temperature to 100 °C from 50 °C did not reduce the viscosity of the polymer solutions. The addition of silica also helps protect biopolymers from microbial and chemical degradation, extending their functional lifespan within the reservoir (Herrera et al., 2017). Based on the results obtained (Fig. 3, 4, and 5), 3 % silica was the optimum concentration because the polymer solution has the highest viscosity at higher shear rate, even with 5 % salinity, and at heated up to 100 °C.

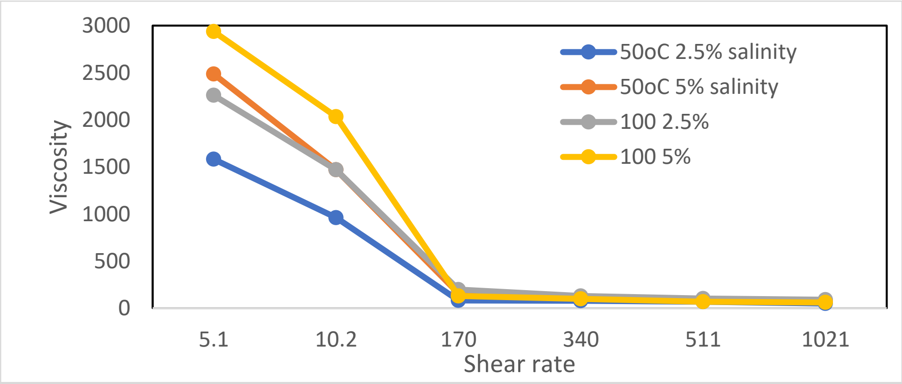

Fig.3. Effect of salinity on the apparent viscosity of the PSPNP at 50 °C for 1 % silica concentration

Source: Author's own data

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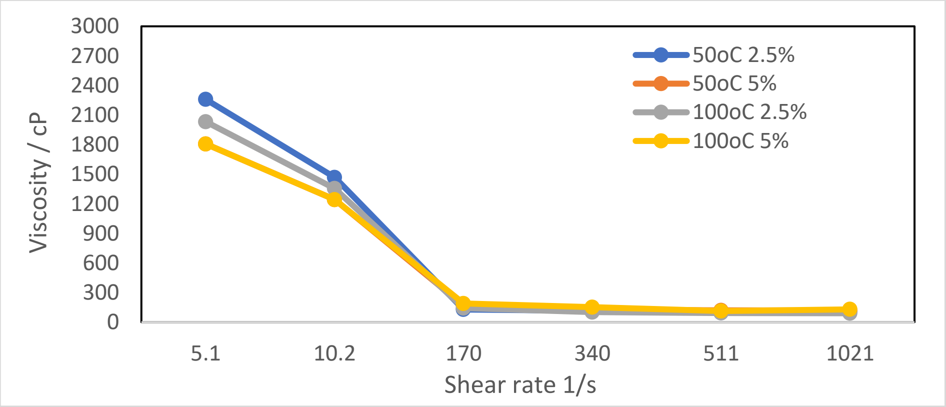

Fig.4. Effect of salinity on the apparent viscosity of the PSPNP at 50 °C for 3 % silica concentration

Source: Author's own data

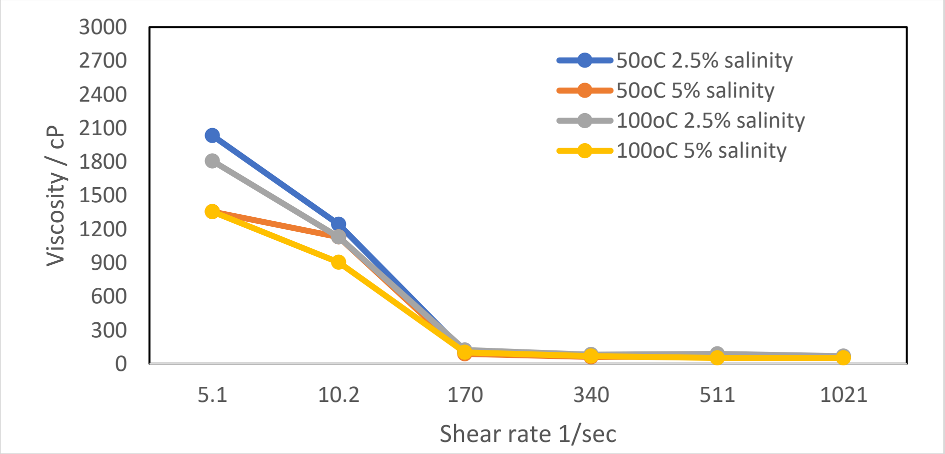

Fig.5. Effect of salinity on the apparent viscosity of the PSPNP at 50 °C for 5 % silica concentration

Source: Author's own data

4. CONCLUSION

With the help of silica, the polymer composite demonstrated high viscosity and significant shear thinning. The presence of silica can improve the rheological properties of biopolymer solutions, making them more stable under reservoir conditions such as high salinity and temperature. Salinity had no major impact on the biopolymer due to its non-ionic composition. The PSPNP demonstrated temperature stability up to 100 °C and maintained viscosity with increasing shear rate. Based on the results obtained, 3 % silica was the optimum concentration to be incorporated in the polymer solution.

https://doi.org/10.24191/mjcet.v8i2.8543

5. ACKNOWLEDGEMENTS/FUNDING

The authors would like to acknowledge the support of the Faculty of Chemical Engineering, Universiti Teknologi MARA, Shah Alam, Selangor, Malaysia, for providing the facilities on this research.

6. CONFLICT OF INTEREST STATEMENT

The authors agree that this research was conducted in the absence of any self-benefit, commercial or financial conflicts, and declare the absence of conflicting interests with the funders.

7. AUTHORS' CONTRIBUTIONS

Muhammad Afiq Iqmal Mohd Yunan: Conceptualisation, methodology, formal analysis, investigation, and writing-original draft; **Norin Zamiah Kassim Shaari**: Conceptualisation, supervision, writing- review and editing, and validation.

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