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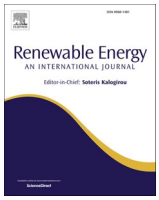

Enhancing microbial fuel cell performance using ceramic additive as biomedia

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ABSTRACT

The ever-increasing pollution on our planet demands the development of clean technologies. Microbial fuel cell (MFC) is one technology with multiple benefits, including, but not limited to, wastewater treatment, bioelectricity generation, heavy metal toxicity reduction, and fertiliser production through catholyte formation. *In-situ* catholyte production in MFCs requires more research to understand how it can be tailored for biofertiliser and bioelectricity production. This study aims to produce better quality catholyte and observe its correlation with bioelectricity generation using ceramic additives known as biomedia. To evaluate its effect on catholyte electrosynthesis and concomitant production of bioelectricity, a mix of fine-grained ceramic additives was introduced to the anolyte. Over the course of the experiment, it was observed that the synthesis of catholyte in MFCs containing biomedia, was 35 % higher in volume compared to that of the MFCs without biomedia, and the electrical conductivity increased to a maximum of 15.72 mS/cm with pH 11.23 with elements such as aluminium being removed from the wastewater in the anode. The current and power generation were also significantly higher in biomedia-MFCs, which suggests its correlation with better quality catholyte. The novel approach of using low cost ceramic additives to amend anolytewas demonstrated as an effective strategy to enhance catholyte production combined with high electricity generation in MFC.

1. Introduction

Microbial fuel cells (MFCs) offer an exceptional choice for combined renewable energy and wastewater treatment. Electro-active microorganisms (EAM) are the key component of MFCs with the capacity to extract chemical energy from biodegradable waste and transform it into electrical energy [1]. Commercialisation of MFCs depends on the optimisation of its constituent components including anode [2], cathode [3] and membrane material [4], microbial community and substrate [5], reactor design configuration and scale [6]. Studies have reported that the membrane type accounts for ca. 40–60 % of the MFC's overall cost [7,8], limiting its practical applicability (Rozendal et al., [9]). Several low-cost membranes with significant output have been tested in MFCs that include eggshell [10]; nylon and glass fibre filter (Zhang et al., [11]); polyester cloth [12] amongst others. An outstanding material, widely tested as a membrane, with the benefits of low cost and low maintenance is the ceramic/terracotta based membrane [13,14].

In MFCs, the biofilm is the critical component, wherein microorganisms adhere to the anode electrode and facilitate the transfer of electrons onto it [15,16]. Maintaining the integrity of the biofilm over the anode is crucial for both the long-term operation of MFC and increased electricity output [17]. Introducing a new material into the anode to maintain biofilm integrity, can be considered as a good improvement strategy. This can enhance electro-activity in the anode and help produce high power output. However, the distribution of the material is required to be uniform and in contact with the electrode. Previous studies have used different types of packing materials/additives to enhance the microbial activity in MFCs including the use of granular activated carbon [18], granulated graphite [18], gravel [19] and polypropylene ([20] Shen et al. [21] used lignite activated coke as packing material in MFC that resulted in enhanced phenolic wastewater treatment along with high power generation compared to that of the MFC operated without the packing material. Packing material/additives helps in increasing the surface area for the enhanced microbial activity, yet there is a substantial drawback associated with it. Tan et al. [20] suggested that packing material might also lead to clogging issues, eventually resulting in mass transfer limitations in MFC, thereby increasing the resistance and reduced MFC performance.

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Consequently, materials with high porosity, that do not clog while providing a greater surface area for microbial activity need to be further investigated.

In addition, the oxygen reduction reaction (ORR) at the cathode, significantly impacts the overall MFC output. ORR is the main reaction in cathode, wherein the electrons transferred from the anode electrochemically reduce the terminal electron acceptor [22]. Oxygen being the most readily available and least expensive, is frequently used as the terminal electron acceptor [23]. The reaction follows equation (1), which is the intermediate step (2-electron pathway) prior to the final step of water production (4-electron pathway). With slow kinetics of ORR, especially in the absence of a catalyst, the reaction generally follows the 2-electron pathway, accumulating H_2O_2 and increasing the pH [24].

$$O_2 + 2H^+ + 2e^- \leftrightarrow H_2O_2 \quad (1)$$

$$H_2O_2 + 2H^+ + 2e^- \leftrightarrow 2H_2O \quad (2)$$

Electricity generation in MFCs gives rise to electro-osmotic drag, which causes water molecules and ions to move from the anode to the cathode. Previous research indicated that this mechanism may function as a cathodic restriction and can be detrimental to the ability of MFCs to produce power [25]. Water movement in the cathode may cause overflow, which impacts the ORR by preventing oxygen from reaching the catalytic site and so hindering the MFC performance. Other studies however, provided a different view, indicating that with proper management, this water can be utilised to enhance MFC performance and facilitate higher electricity production [26,27]. Furthermore, this catholyte could be utilised for carbon capture [26], sodium recovery [28] and caustic potash production [27].

This study aims to evaluate the feasibility of using a combination of fine-granulated ceramics as a filling material that can function as biomedia in the MFC anode, enabling the growth of electroactive organisms from within. Ceramic is considerably low cost and has high bio-affinity and large surface area, which can lead to higher microbial electroactive activity in the anode. The impact of biomedia on mass transfer resistance, electroosmosis (catholyte production), and electricity generation is examined. Physicochemical and elemental analysis of the produced catholyte was done to understand the correlation of biomedia, catholyte and electricity generation.

2. Materials and methods

2.1. MFC design, construction, and inoculation

The study uses MFCs with an exterior anode and an inner cathode composed of 12.3 % porosity terracotta cylinders (which served as the chassis and membrane) [29]. The cylinders measured 5 cm in height, 2.2 cm diameter, with a thickness of ca. 3 mm. The cylinders were placed inside a plastic container with working anode volume of 120 mL. Carbon fibre veil with a total macro-surface area of 30 × 30 cm^2, and a carbon loading of 20 g/m^2 (PRF Composite Materials, Dorset, UK) was used as the anode, wrapped around the outside of the cylinder. The cathode electrodes consisted of a mixture of activated carbon (G. Baldwin & Co, 80 gr) and a 60 % polytetrafluoroethylene (PTFE) solution (Sigma-Aldrich) dispersed over a stainless-steel mesh support. The method of preparing the cathode was as described in Ref. [26]. The cathode electrodes were cut with a geometric area of 4*4 cm^2 and introduced into the internal part of the ceramic cylinders. The cathode electrodes were manually pushed inside the cylinders, pressed against the wall for better contact, and kept open to air. Ni-Cr wire was employed for both cathode and anode connections. Assembled MFCs were then inoculated with 1:1 part raw activated sludge and 1 % tryptone, 0.5 % yeast extract and 20 mM sodium acetate mixture, in equal amounts of 50 mL. The anolyte was replenished daily by withdrawing 10 mL and adding 10 mL of fresh feedstock (1 % tryptone, 0.5 % yeast extract, and 20 mM sodium acetate) to prevent biofilm starvation. Triplicates of MFCs were used for conducting the experiments.

2.2. Filling material/ceramic biomedia

Patented ceramic spheres of different diameters ranging from 3 mm–40 mm developed by research carried out by Asian Institute of technology (AIT) for commercial wastewater treatment were supplied by Siam Cement Group (SCG). These granular ceramics were grounded further reaching almost a fine powder form and the mixture of both powder and granular ceramics was used as “biomedia” in the anode of MFC (Fig. 1). The term “biomedia” is used to describe the function of these ceramic spheres as host material for microbial growth, which enables continuous growth from the inside, especially given the material’s chemical composition (see EDX analysis, Fig. S1).

2.3. Electrochemical characterisation

The open circuit voltage (OCV) and closed-circuit voltage (CCV) of the control MFC without biomedia (MFC-C) and the biomedia MFCs (MFC-B) were recorded on a daily basis using a high-precision multimeter (Fluke 287/289 Digital Multimeter). A 330 Ω resistor was used to load these MFCs and the value was determined from previous studies with the same ceramic cylinders as membrane. The current and power output were calculated based on Ohm’s law i.e. I=V/R, where V is the voltage in closed circuit and R is the resistance value and I is the current. Power is calculated as P=V*I. Power density was calculated as the absolute power divided by the surface area of the anode square meters (m^2) or by the volume of the anodic fluid in m^3 [30]

Polarisation was performed, once it was visually evident that the biofilm begun to mature in all the MFCs, after 6 days from the start of the experiment. Polarisation experiments were carried out by varying the external resistance from 10000 Ω to 10 Ω using a decade resistance box (ELC, DR06) every 3 min. Internal resistance was calculated from the slope of the V-I curves. Maximum power transfer was determined from the P-I graph and in general this occurred when the voltage at load was half of open circuit voltage.

2.4. Physicochemical and elemental testing

In all the MFCs, catholyte was occasionally collected with a syringe just before flooding to test its quality. Conductivity and pH were measured using a Hanna Bench Meter (HI5522). A crystal deposit was formed when the collected catholyte was allowed to evaporate. The elemental composition of the catholyte was ascertained using energy dispersive X-ray spectroscopy (EDX) (Zeiss Sigma 500 VP FESEM). Energy dispersive X-ray (EDX) analysis was also used to investigate the chemical composition of the ceramic biomedia. Scanning Electron Microscopy (SEM) (Zeiss Sigma 500 VP FESEM) was used to test the morphological composition of microorganisms over the anode electrode from MFC-B.

3. Results and discussion

3.1. Morphological analysis

SEM analysis was used to test the microorganism’s dominance on the carbon veil electrode in the presence of biomedia. The porous structure of the ceramic biomedia as found in SEM analysis is given in Fig. S2. Over the carbon veil electrode, biomedia was clearly visible (shown by arrow in Fig. 2). A healthy and rich microbial community as a significant abundance of extracellular polymeric substances (EPS) on the carbon veil electrode was seen in presence of ceramic biomedia (Fig. 2). EPS on the electrode surface promotes a variety of metabolic interactions in addition to improving microbial adherence, suggesting the development of a robust and active microbial ecosystem. This microbial diversity is

Fig. 1. Ceramic spheres used as biomedia.

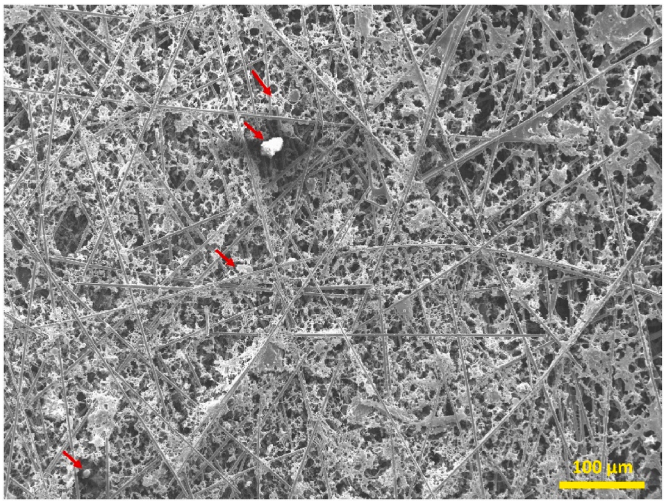

Fig. 2. SEM image of carbon veil electrode from MFC-B showing rich microbial diversity in the presence of ceramic biomedia.

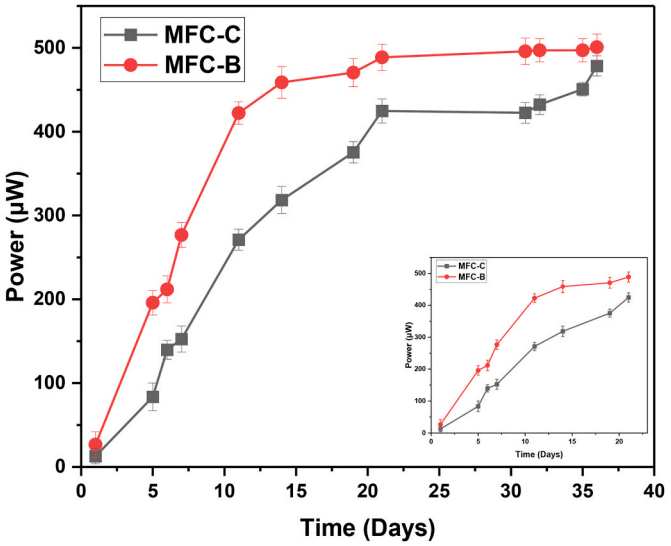

Fig. 3. Temporal power output performance from the two test conditions, whereby MFC-B is with biomedia and MFC-C is the control without biomedia. Experiments were run in triplicates however data from the biomedia set are from n = 2, since one of the replicate units had continuous connection problems.

essential for effective electron transfer activities, which support the functionality and stability of bioelectrochemical systems.

3.2. *Effect of biomedia on bioelectricity generation*

To obtain a stable open circuit voltage, all the MFCs were run in open circuit for 24 h. The average open circuit voltage in MFCs with biomedia (MFC-B) was 629 mV ± 5, which was slightly higher than that produced in MFCs without biomedia (615 mV ± 6). Then, all the MFCs were loaded using a 330 Ω resistor. After 2–3 days under closed circuit conditions, the MFC output began to stabilise. Once a steady closed-circuit voltage was obtained, power was calculated for each of these MFCs. Fig. 3 illustrates the increase in power generation that occurred when these MFCs were operated in closed circuit for 21 days. It is evident that the power output from the MFC-B was consistently higher from the start; within 10 days of the experiment, >400 µW of power was generated, whereas 400 µW in MFC-C could only be reached by the end of the 19th day. The biomedia-introduced MFC had significantly shortened its startup time and continued to produce higher electricity than the control MFC.

3.2.1. *Polarisation*

Polarisation runs were carried out on day 6, 13 and 36 of the experimental period and Fig. 4 shows the power and polarisation curves of MFC-B and MFC-C. On day 6, maximum power transfer in MFC-B (3.68 mW/m^2) was 272 % greater than that of MFC-C (0.99 mW/m^2). On day 13, maximum power transfer in MFC-B (9.8 mW/m^2) was 100.4 % higher than MFC-C (4.89 mW/m^2), and on day 36, maximum power transfer in MFC-B (13.69 mW/m^2) was 28.2 % higher than MFC-C (10.68 mW/m^2). MFC-B consistently had higher maximum power transfer, indicating effective substrate utilisation and microbial activity in the presence of biomedia.

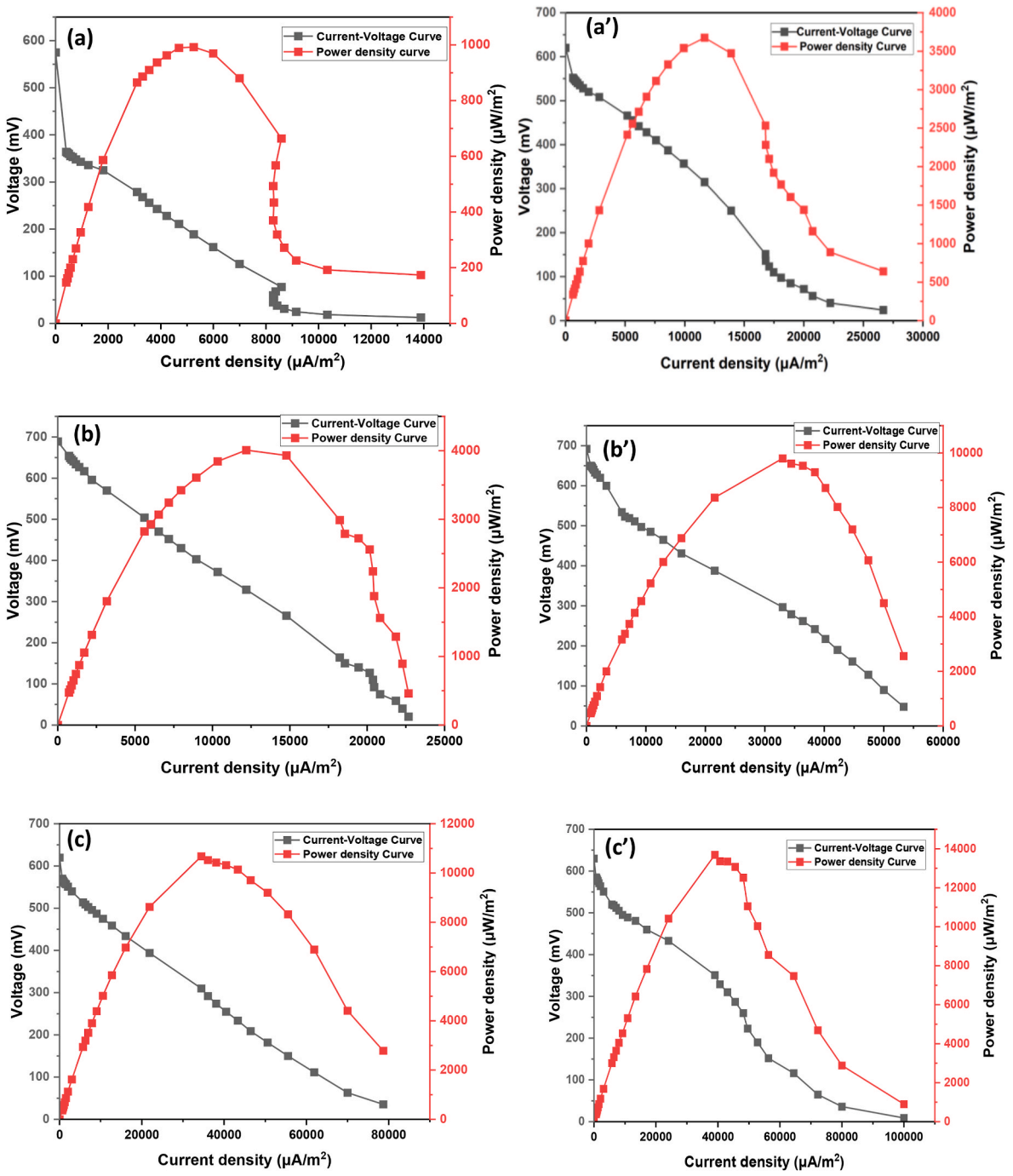

Fig. 4. Polarisation results of control MFCs (without biomedia) on [(a) day 6 (b) day 13 and (c) day 36] and biomedia carrying MFCs (MFC-B) on [(a') day 6 (b') day 13 and (c') day 36].

Internal resistance measured in MFC-B was initially 244 Ω (day 3); this decreased to 66 Ω after 33 days of running, which was lower than that of MFC-C, (375 Ω on day 3 and 73 Ω on day 36). Resistance was noticeably reduced in MFC-B, indicating a greater generation and utilisation of electrons in the production of electricity. This suggests that the porosity of ceramic biomedia assisted in lowering the transport resistance in the MFC, and the use of ceramic as packing material/biomedia has helped to reduce the internal resistance.

In addition to acting as a space for bacterial proliferation and dense biofilm generation, it is assumed that the pores of the ceramic biomedia reduced transport resistance within the MFCs, facilitating mass transfer. Mass transfer limitation in MFCs has been found to significantly limit the MFC output [31,32]. Various packing/filling/additive materials and media have been employed in previous studies. Mu et al. [33] used different kinds of filler materials to simultaneously remove chromium and produce electricity in constructed wetland-MFC. They discovered that volcanic rock functioned as the ideal biomedium, supporting high organic matter elimination and high electricity. This was confirmed by the maximum electroactive activity in the presence of volcanic rock. Zhang et al. [11] used activated carbon and graphite material as biocarrier in anode and found reduced start-up time, high COD removal and increased electrical output in MFC. However, it has also been observed that such kind of materials might end up clogging the anodic part [20]. On the other hand, using this ceramic-based, high bioaffinity biomedia, has led to a kind of self-cleaning effect that gradually dissolves solids, increases the conductivity of the medium, and is eventually utilised by

the microorganisms. Consequently, this has prevented any obstruction to the anode medium.

3.3. Role of biomedia on catholyte (quantity, pH and EC) and trend of catholyte production with current generation

The electroosmosis-based catholyte generation in MFCs running with and without biomedia was investigated (Fig. 5). On the surface of the cathodes, droplets were seen to develop during the first week, however only from the 7th day onwards, a quantifiable amount of catholyte was observed. There was a continuous trend of higher catholyte accumulation in MFC-B compared to that of MFC-C during the experiment. The catholyte quantity was measured up to the period of 21 days. The total catholyte collected by the end of 21 day was 13.8 mL, 35.3 % higher than that of MFC-C (10.2 mL). As can be seen from Fig. 5 below, the catholyte volume is also a function of time, which increases as time progresses. Earlier studies have reported that the volume of accumulated catholyte is directly proportional to the current [28] which suggests that catholyte accumulation is a function of MFC performance. Rismani-Yazdi et al. [25] suggested that cathode flooding may impair MFC performance however, earlier studies has indicated that, when appropriately managed, accumulation of catholyte may contribute to increased electricity output [27,34]

Biomedia contributes to increasing the surface area available for microbial activity, which in turn enhances charge transfer. The present study has also found a significant high correlation of catholyte accumulation and current generation in MFC (Fig. 6). The growth and accumulation of microorganisms is helping in higher electroosmosis, leading to higher catholyte synthesis.

The collected catholyte was gathered and examined for its quality by measuring its pH and EC. Fig. 7 represents pH of the catholyte generated from MFC-B and MFC-C over the experimental period. There was a continuous increase of pH observed in the biomedia MFC. The pH started from 10.07 in first week and increased to 11.27 by the end of day 36 in the case of MFC-B, whereas it only increased from 9.58 to 10.37 in MFC-C. The addition of biomedia in the anolyte, appears to produce a more alkaline catholyte. Earlier studies have confirmed that higher microbial cell activity will generate more current leading to increase the pH levels [34]. The cathode half reaction leads to a higher production of OH^- groups that accumulated on the cathode and helped to raise the pH. This is because of the active pathway of ORR, particularly in the absence

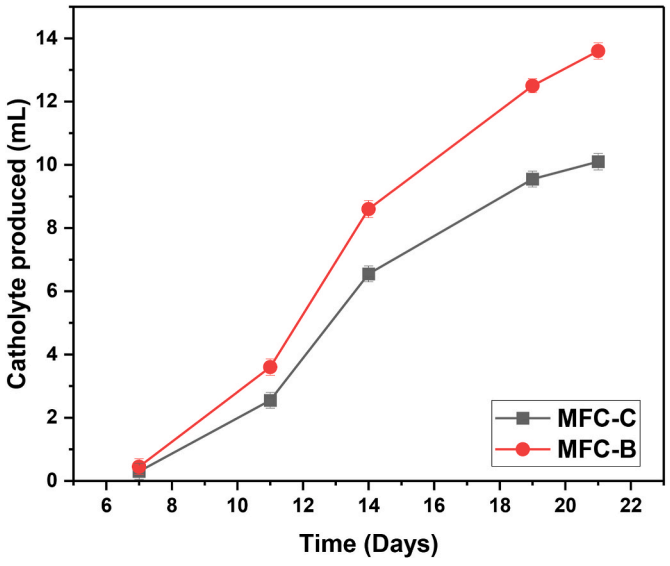

Fig. 5. Catholyte volume synthesised over the period of 21 days. Data shown are from the two triplicates, with biomedia being n = 2 for the sake of consistency.

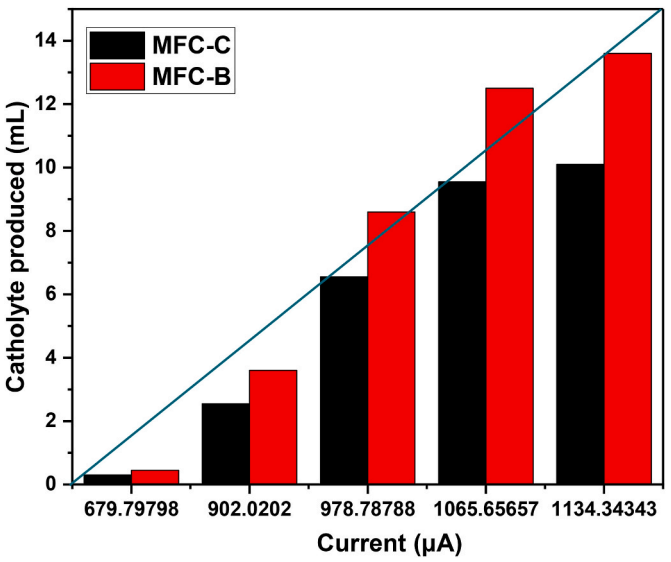

Fig. 6. Trend between catholyte production and current at 330 Ω over a period of 21 days.

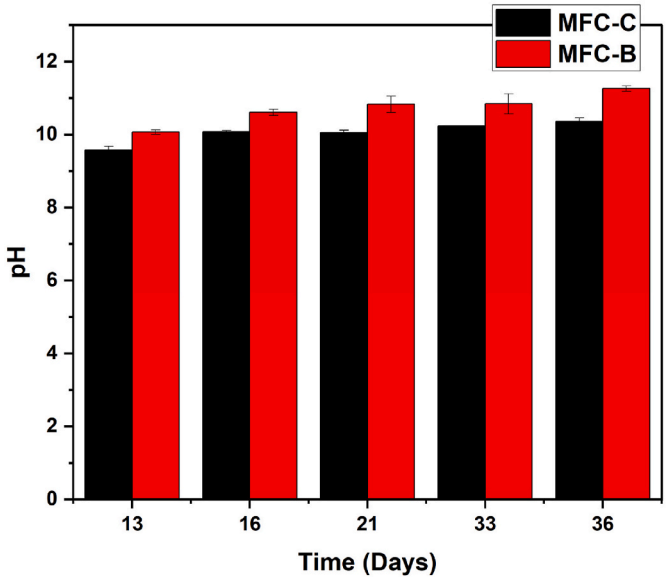

Fig. 7. Gradual pH increase of catholytes obtained from MFC-C and MFC-B.

of any catalyst as suggested by Babauta et al. [24]. The findings of Gajda et al. [28] and Popat et al. [35] also corroborated with the former study.

The conductivity of catholyte in MFC-B started from 5.82 $mScm^{-1}$ and reached to 15.72 $mScm^{-1}$, found to be higher than MFC-C that started with 5.57 $mScm^{-1}$ and could reach to 11.27 $mScm^{-1}$ by the end of experiment (36 days) (Fig. 8). The transport of ions is highly dependent on the current generation capacity of MFC, which is what is being observed in the present study. This also reduces the chances of biofouling on the cathode, as higher catholyte volume is leading to dissolved salt from the electrode. This is why earlier studies have suggested that this catholyte can serve as an excellent agent for disinfectant [26] instead of using external disinfectant for cathode chamber [36].

EDX spectra of catholyte obtained from MFC-C and MFC-B are shown in Fig. 9 (a) and (b), respectively. The elemental analysis of the catholyte has shown presence of numerous elements in MFC-B as well as MFC-C. However, the presence of certain elements especially Na^+ and K^+ were found in more abundance in the catholyte collected from the biomedia MFCs (Fig. 10). This suggests that the MFCs with biomedia,

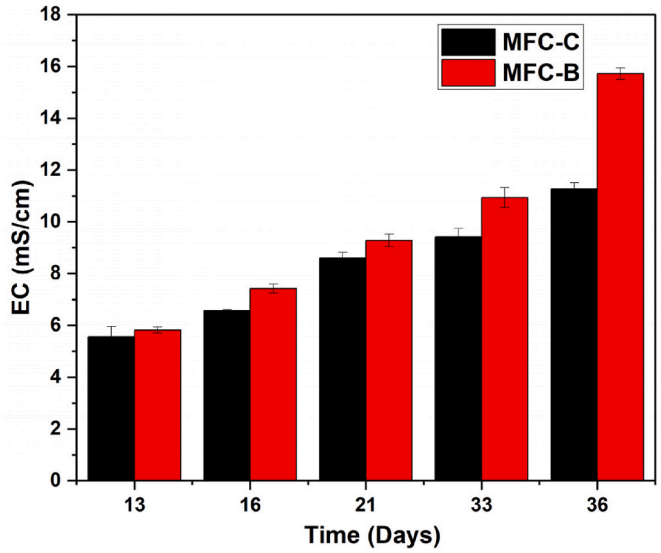

Fig. 8. Gradual conductivity increase of catholyte samples obtained from MFC-C and MFC-B.

were more actively, electroosmotically dragging the cations commonly found in wastewater, to accommodate for the higher charge transfer, in line with the law of electroneutrality. Due to its high pH, high conductivity, and presence of positive ions, the catholyte produced in the presence of biomedia may be used as a fertiliser or disinfectant. EDX spectra of the catholyte has also shown that the carbon content from MFC B is 11.1 % which is lower than that of obtained from MFC-C (23.1 %). This accounts for the increased anolyte treatment efficiency in MFC-B, which results in higher-quality catholyte with lower carbon content.

Interestingly, aluminium (Al), commonly used in wastewater treatment for coagulation, was only seen in the catholyte of the MFCs without biomedia and was not detected in the catholyte of the MFCs with

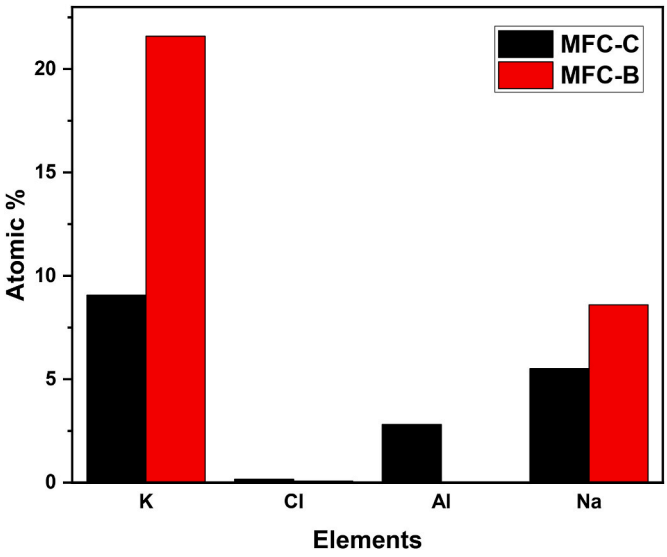

Fig. 10. Comparative composition of elements presents in the evaporated catholyte obtained from MFC-C and MFC-B.

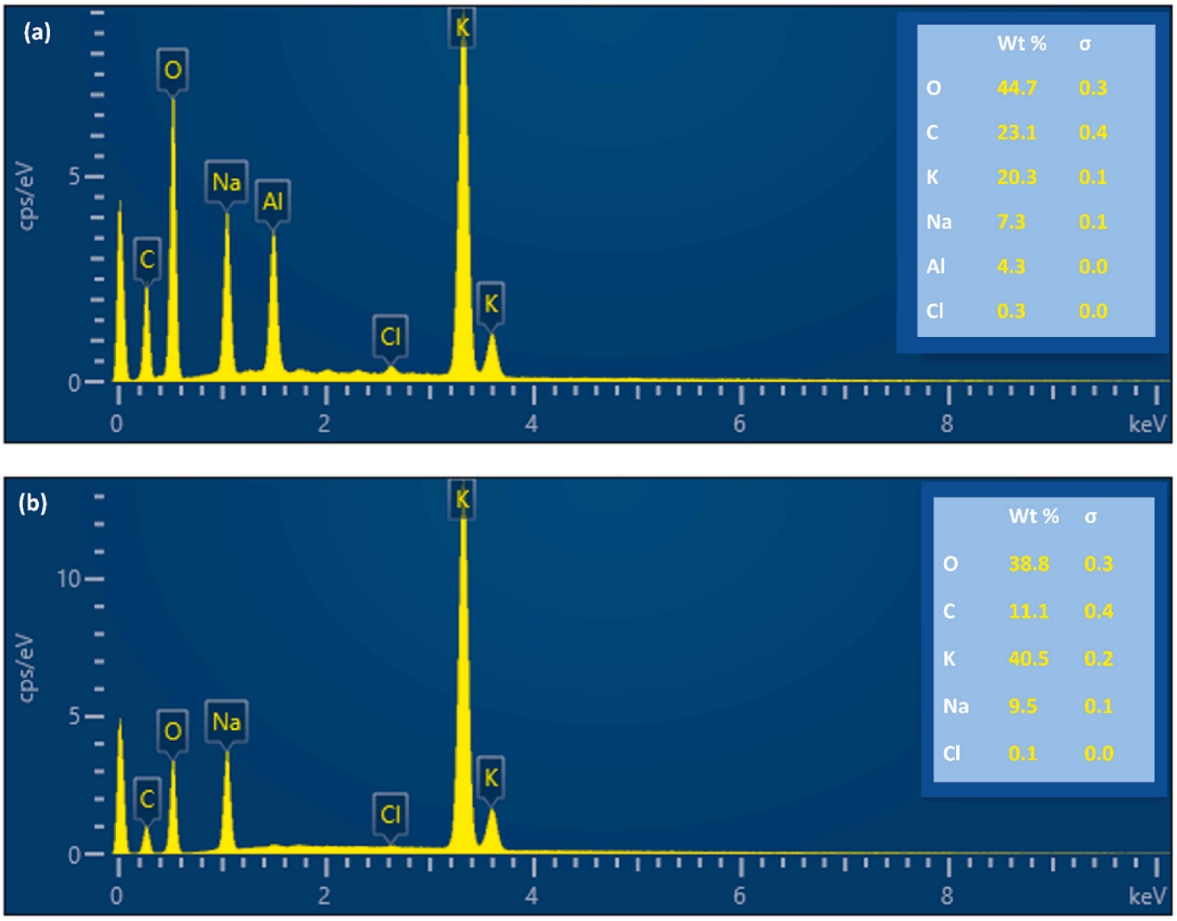

Fig. 9. EDX spectra of the evaporated catholyte obtained from (a) MFC-C and (b) MFC-B.

biomedia. This suggests that either the biomedia was actively adsorbing Al or the biomedia-supported microbial communities were possibly assimilating Al as a co-metabolite, therefore removing it from wastewater in the anode. This is also corroborated by Purwanti et al [37] who observed removal of Al from the wastewater and concluded the role of biosorption and bioaccumulation by two different isolated bacteria. Active adsorption of Al by a certain kind of carriers such as activated carbon from rice hull [38], active carbon with different grading [39], and biopolymers [40] from various effluent types has already been effectively investigated in the aforementioned studies.

This could be an interesting area of research to investigate, also, for other elements in the presence of biomedia in MFCs. It is evident that biomedia plays a crucial role in generating more power while also lowering anode toxicity and creating high-quality catholyte. SEM images of the anode electrodes (Fig. 2) seem to support this finding.

4. Conclusions

This study confirmed that the use of biomedia was highly effective in enhancing the overall electrical output of MFCs. The start-up time was reduced, and maximum power output was reached quicker in MFC-carrying biomedia. The maximum power density was more than 250 % higher in biomedia-based MFCs compared to that of MFCs operated in the absence of biomedia. This difference reduced however, and was shown to be around 28 % higher even by the end of the experiment (day 36). It was also observed that biomedia facilitated higher amounts of catholyte, which was directly proportional with high current production. The catholyte was also found to be better in quality in the case of biomedia-based MFCs. The pH ranged from 10.07 to 11.27, whereas in the MFCs without biomedia, the pH variation was 9.58–10.37. Highest conductivity of 15.72 $mScm^{-1}$ was also achieved in biomedia based MFC. Catholyte volume, pH and conductivity increased with time, which suggests a correlation with MFC biofilm maturity and higher current generation. Elemental analysis showed the higher concentrations of Na^{+} and K^{+} in the catholyte generated from MFCs carrying biomedia. The work suggests that further research on filler materials that function as a biohouse for microbes in the MFC anode should be conducted. These materials can be utilised to boost the surface area available for the growth of microorganisms and lower the resistance brought on by mass transfer constraint in MFC.

CRediT authorship contribution statement

Aradhana Singh: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Data curation. **Ioannis A. Ieropoulos:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.renene.2025.122738.

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