RGC Ref. No.:
UGC/FDS16/P02/20
(please insert ref. above)

RESEARCH GRANTS COUNCIL
COMPETITIVE RESEARCH FUNDING SCHEMES FOR
THE LOCAL SELF-FINANCING DEGREE SECTOR

FACULTY DEVELOPMENT SCHEME (FDS)

Completion Report
(for completed projects only)

Submission Deadlines:
1. *Auditor's report with unspent balance, if any: within **six** months of the approved project completion date.*
2. *Completion report: within **12** months of the approved project completion date.*

Part A: The Project and Investigator(s)

1. Project Title

Investigation of metal oxide-based nanofiber as cost-effective catalyst for biodiesel synthesis

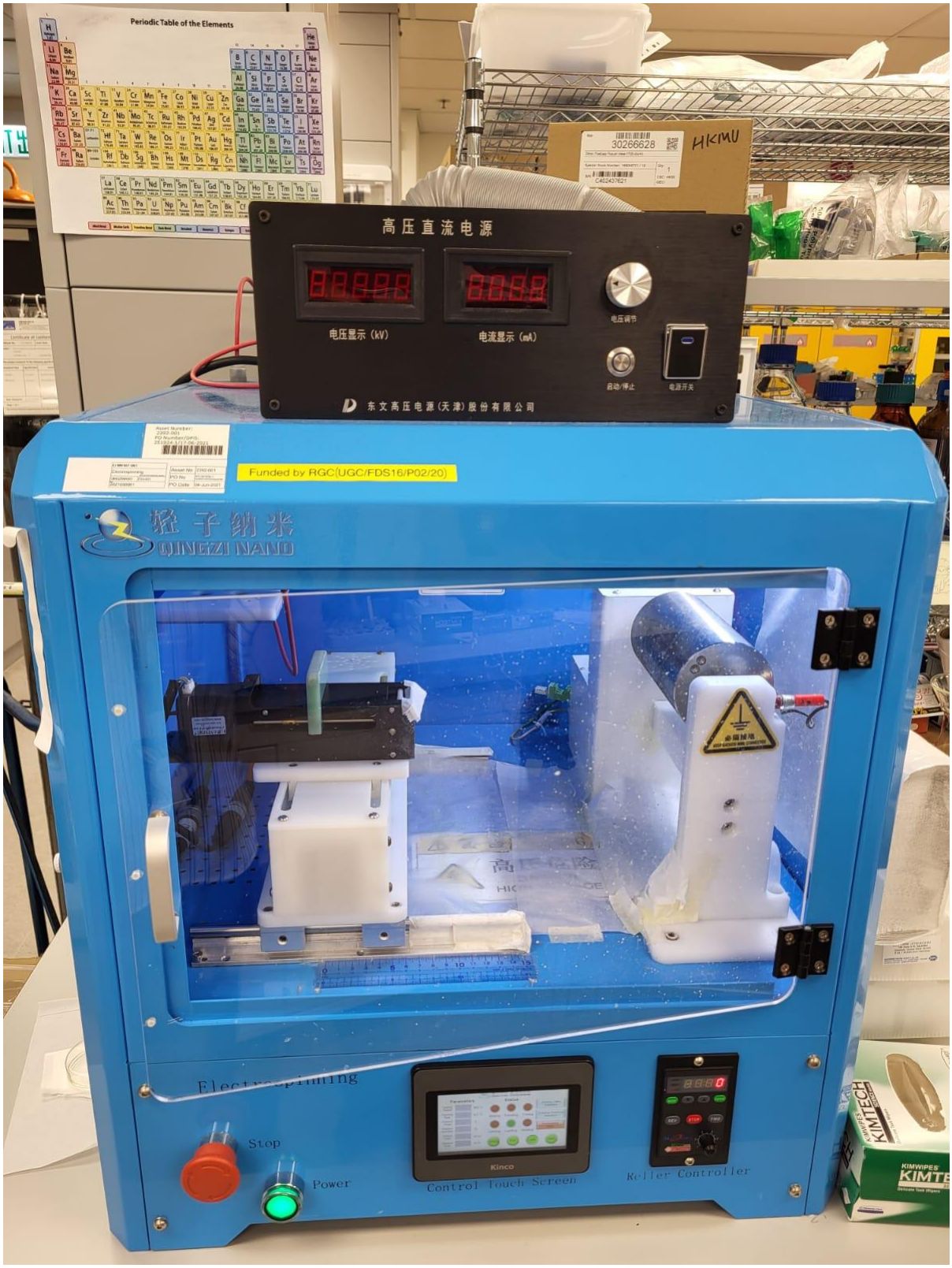
2. Investigator(s) and Academic Department(s) / Unit(s) Involved

Research Team	Name / Post	Unit / Department / Institution
Principal Investigator	NG Christie Morgan Ching-man / Senior Lecturer	Department of Applied Science, School of Science and Technology, Hong Kong Metropolitan University
Co-Investigator(s)	N/A	N/A
Others	N/A	N/A

3. Project Duration

	Original	Revised	Date of RGC / Institution Approval *(must be quoted)*
Project Start Date	01 Jan 2021	N/A	
Project Completion Date	31 Dec 2023	30 June 2024	08 Nov 2022
Duration *(in month)*	36	42	08 Nov 2022
Deadline for Submission of Completion Report	31 Dec 2024	30 June 2025	08 Nov 2022

4.4 Please attach photo(s) of acknowledgement of RGC-funded facilities / equipment.



Part B: The Final Report

5. Project Objectives

5.1 Objectives as per original application

1. To synthesize and characterize two types of metal oxide nanofibers of different fiber diameters which will be made from two types of polymers by electrospinning
2. To investigate the catalytic performance of different types of metal oxide nanofibers on biodiesel synthesis from inedible crude oil, used cooking oil and grease trap waste

5.2 Revised objectives

Date of approval from the RGC: N/A

Reasons for the change: N/A

1.

2.

3. ....

5.3 Realisation of the objectives
(Maximum 1 page; please state how and to what extent the project objectives have been achieved; give reasons for under-achievements and outline attempts to overcome problems, if any)

Objective 1: Zirconia and tungsten oxide nanofibers in two polymers, PVA and PAN, have been successfully synthesized via electrospinning. Both zirconia and tungsten oxide nanofibers electrospun from PVA had a smaller average fiber diameter (in nm scale), whereas PAN had a larger average fiber diameter (in micron scale) and the fibers are visible to naked eyes during the process. However, there were some difficulties encountered when modifying the fiber surface with acid. In the first attempt, all fibers formed with Zr and W precursors dissolved upon the addition of sulphuric acid. Therefore, the second attempt was to form zirconia and tungsten oxide nanofibers first, then surface modification was carried out. The zirconia fibers surface could not be modified due to the formation of crystalline zirconia [1], where surface modification must be carried out before the formation of oxides. Finally, S-ZrO_2 and WO_3 nanoparticles was suspended into PVA and PAN. However, the fibrous structure collapsed upon high temperature calcination. Further analysis indicated the nanoparticles were suspended unevenly in the polymer solution, and failed to form a fibrous structure during electrospinning.

Objective 2: Biodiesel conversion catalyzed by the nanofibers and nanoparticles synthesized in objective 1 have successfully conducted on a system mimicking waste oil. The product yield was initially studied with ^{1}H NMR. However, the purchase of NMR solvent required license, and it took half a year to complete the whole purchasing and import of NMR solvents. Then the next difficulty was the occasional malfunctioning of NMR instrument since late 2023. While the engineer was fixing the NMR, the project team switched to GC for data analysis. A wrong GC standard was delivered by the

supplier, and it took about a month's time to re-deliver the correct GC standard. The project team experienced difficulty with GC since the column was different to the one reported in literature, so fine tuning of the GC condition was required. Despite the GC calibration curve was finally constructed, the GC instrument was often occupied by undergraduate teaching that uses a different column as our study. Frequent changing of GC column is required, and together with the university's busy teaching schedule, the samples could not be analyzed in time. Near to the project end date (June 2024), the engineer confirmed the NMR could not be fixed. Since NMR was the most efficient method because data can be obtained instantly and calculations can be done directly, the project team seek collaboration with PolyU for their support in ^{1}H NMR analysis. However, there was extensive delays in the data analysis submitted to PolyU due to the busy schedules of the institute's own teaching and research purposes. Thus, all conversions have been conducted, and preliminary data has been collected. The last batch of data analysis is still underway.

[1] Sun, Y., Ma, S., Du, Y., Yuan, L., Wang, S., Yang, J., Deng, F., Xiao, F.S. Solvent-Free Preparation of Nanosized Sulfated Zirconia with Brønsted Acidic Sites from a Simple Calcination. *J. Phys. Chem. B* **2005**, *109*, 2567–2572. https://doi.org/10.1021/jp046335a

5.4 Summary of objectives addressed to date

Objectives *(as per 5.1/5.2 above)*	**Addressed** *(please tick)*	**Percentage Achieved** *(please estimate)*
1. To synthesize and characterize two types of metal oxide nanofibers of different fiber diameters which will be made from two types of polymers by electrospinning	✓	100%
2. To investigate the catalytic performance of different types of metal oxide nanofibers on biodiesel synthesis from inedible crude oil, used cooking oil and grease trap waste	✓	100%

6. Research Outcome

6.1 Major findings and research outcome
(Maximum 1 page; please make reference to Part C where necessary)

Objective 1: Different morphologies and length of zirconia and tungsten oxide nanofibers was formed depending on the PVA and PAN concentration. When using PVA as the support, zirconia (in 10.5 wt% PVA) and tungsten oxide (in 10 wt% PVA) nanofibers with smooth fibrous morphology was retained after the polymer was removed at high temperature calcination The average fiber diameter was 60 nm and 300 nm respectively. While at other PVA concentrations, shorter fibers were formed. When PAN was used as the support, the average fiber diameter of both metal oxides was found to be much larger than using PVA, with an average fiber diameter of 0.6 – 0.8 μm after the polymer was removed upon calcination. Also, the fiber morphology was rough. The surface modification of the nanofibers was unsuccessful (as stated in 5.3), so the suspension of S-ZrO_2 and WO_3 nanoparticles was conducted. With the suspension of nanoparticles in PVA and PAN, the fibrous structure broke apart upon calcination and only S-ZrO_2 and WO_3 nanoparticles were retained.

Objective 2: Both ZrO_2 and WO_3 nanofibers only showed very low yield on biodiesel conversion, below 5% conversion for ZrO_2 and ca. 2% conversion for WO_3 was achieved after 2 h at 140°C, 8 wt% catalyst loading, 1:20 oil-to-methanol ratio. The low conversion was caused by a number of reasons. It was observed that a certain degree of oil was absorbed by the composite metal oxide nanofibers as they were mixed in the reaction vessel. When the reaction mixture was heated, the composite nanofibers floated on the reaction mixture surface, further reducing the contact area between oil with the reaction sites on the catalyst. Moreover, since only small amount of nanoparticles was suspended in the nanofibers, it was proposed minimal reaction sites were available for reaction. Further increasing the amount of S-ZrO_2 and WO_3 nanoparticles suspended in polymer would lead to frequent blockage of needle and fibers could not be formed. As the fibrous structure could not be retained upon high temperature calcination, catalytic study of S-ZrO_2 nanoparticles on biodiesel synthesis was further studied using microwave as the heating source. S-ZrO_2 nanoparticles was found to have a surface area of ca. 48 m^2 g^{-1}, total pore volume of ca. 0.2 cm^3 g^{-1}, and an average pore sizes of 3-9 nm. Due to low conversion catalyzed by WO_3 and the difficulties with data analysis mentioned in 5.3, WO_3 catalyzed biodiesel conversion using microwave as the heating source was not further studied at this stage. Microwave has been reported to be an effective heating source for biodiesel synthesis, and biodiesel synthesis catalyzed by chlorosulphonic acid modified zirconia nanoparticles using microwave as the heating source has not yet been reported to date.

A non-catalyzed reaction at 1:20 oil-to-methanol ratio, 120°C (microwave power 200 W) showed only 0.8% biodiesel yield was achieved after 30 min. When microwave irradiation was used, 15.9% biodiesel yield was achieved in 30 min with 4 wt% catalyst at the same oil-to-methanol ratio and reaction temperature. A similar reaction was conducted with non-modified zirconia under the same reaction conditions and microwave power, where only ca. 29% biodiesel yield was achieved after 3 h. The presence of sulphate groups on zirconia greatly enhanced the catalytic activity, and S-ZrO_2 nanoparticles showed a promising catalytic activity within 30 min under microwave irradiation in mimicking waste oil transesterification reaction. The optimal reaction condition was determined to be 4 wt% catalyst, 1:20 oil-to-methanol ratio and 160°C reaction temperature using 200 W microwave power.

6.2 Potential for further development of the research and the proposed course of action *(Maximum half a page)*

Our study showed the potential of surface modified zirconia as a promising catalyst

on the conversion of biodiesel using microwave irradiation. Upon determining the optimum reaction condition, future work will be focused on extending the reaction time for achieving a higher biodiesel yield. The reusability of the catalyst is another important factor to investigate. If the catalyst is reusable for a few cycles without major decrease in the biodiesel conversion, this makes the overall process greener and more sustainable. Moreover, a new spherical nanostructure of S-ZrO_2/WO_3 was discovered during the synthesis of ZrO_2 and WO_3 nanoparticles for this project. A manuscript is currently under preparation, once the project team has received the NMR data from PolyU, the manuscript will be ready to submit.

7. Layman's Summary

(Describe in layman's language the nature, significance and value of the research project, in no more than 200 words)

Zirconia continuous nanofibers are fabricated with 10.5 wt% polyvinyl alcohol (PVA) and 10 wt% polyacrylonitrile (PAN) using electrospinning, which is a cost-effective method to synthesize nanofibers. The nanofibers electrospun from PVA has a smooth surface, with an average fiber diameter of 60 nm. In contrast, the nanofibers surface fabricated from PAN is rough, with an average fiber diameter of 0.6 – 0.8 μm. At other polymer concentrations, shorter fibers were formed. The fibrous structure broke apart upon high temperature calcination when S-ZrO_2 nanoparticles were suspended in PVA and PAN. S-ZrO_2 nanoparticles shows a promising catalytic activity in biodiesel conversion under microwave irradiation. Around 29% biodiesel conversion was achieved within 30 min in a system mimicking waste oil composition, with optimized reaction condition of 4 wt% catalyst, 1:20 oil-to-methanol ration and 160°C.

Part C: Research Output

8. Peer-Reviewed Journal Publication(s) Arising Directly From This Research Project

(Please attach a copy of the publication and/or the letter of acceptance if not yet submitted in the previous progress report(s). All listed publications must acknowledge RGC's funding support by quoting the specific grant reference.)

The Latest Status of Publications				**Author(s)** *(denote the corresponding author with an asterisk*)*	**Title and Journal / Book** *(with the volume, pages and other necessary publishing details specified)*	**Submitted to RGC** *(indicate the year ending of the relevant progress report)*	**Attached to this Report** *(Yes or No)*	**Acknowledged the Support of RGC** *(Yes or No)*	**Accessible from the Institutional Repository** *(Yes or No)*
Year of Publication	**Year of Acceptance** *(For paper accepted but not yet published)*	**Under Review**	**Under Preparation** *(optional)*						
		✓		Christie Ng*, Yuen On Fung, Chi Ho Li and Ka-Fu Yung*	Synthesis of composite zirconia nanofiber: a model study in microwave-assisted biodiesel conversion (Submitted to Nanomaterials)	No	Yes [Attachment 1]	Yes	Yes
			✓	Yuen On Fung, Wing Tung Ho, Chi Ho Li, Ka-Fu Yung* and Christie Ng*	Solvent-free synthesis of a novel S-ZrO2/WO3 spherical nanostructures supported on hexagonal plates (Plan to submit to Frontiers in nanotechnology)	No	No	Yes	Yes

9. Recognized International Conference(s) In Which Paper(s) Related To This Research Project Was / Were Delivered
(Please attach a copy of each conference abstract)

Month / Year / Place	**Title**	**Conference Name**	**Submitted to RGC** *(indicate the year ending of the relevant progress report)*	**Attached to this Report** *(Yes or No)*	**Acknowledged the Support of RGC** *(Yes or No)*	**Accessible from the Institutional Repository** *(Yes or No)*
Aug 2023 London, United Kingdom	Synthesis of Nanofibre Catalyst in AI-based Electrospinning Process for Sustainable Biodiesel Production	The Seventh Edition of the World Conference on Smart Trends in Systems, Security and Sustainability (WordS4 2023)	No	Yes [Attachment 2]	Yes	Yes

10. Whether Research Experience And New Knowledge Has Been Transferred / Has Contributed To Teaching And Learning
(Please elaborate)

Analytical instruments used in this project (i.e. SEM, TEM, EDX, XRD and NMR) and electrospinning to synthesize nanofibers were not introduced in any of the undergraduate (UG) courses in Hong Kong Metropolitan University (HKMU). Students have no knowledge on the theory, and do not have any hands-on experience on the operation of these instruments. The skills and knowledge of these instruments have been transferred to 2 students listed in part 11 under UG course SCI S390F Professional Training and Workplace Attachment and "Next Generation Scientist Incubation (NGS-i) Programme". NGS-i programme enhances UG student's research skills by providing 2-year research training.

The concept of biodiesel and its impact on the environment has been introduced to students in the following UG courses: SCI S390F Professional Training and Workplace Attachment, BIOL S401F Contemporary Biology Development, CHEM S306F Advanced Environmental Analysis and SCI S411F Environmental Health and Safety.

11. Student(s) Trained
(Please attach a copy of the title page of the thesis)

Name	**Degree Registered for**	**Date of Registration**	**Date of Thesis Submission / Graduation**
	Bachelor of Science (Hons) in Life Sciences	Undergraduate student	Year of gradation 2022
	Bachelor of Science (Hons) in Testing Science and Certification	Undergraduate student	Year of gradation 2025

12. Other Impact

(e.g. award of patents or prizes, collaboration with other research institutions, technology transfer, teaching enhancement, etc.)

Same as part 10

13. Statistics on Research Outputs

	Peer-reviewed Journal Publications	Conference Papers	Scholarly Books, Monographs and Chapters	Patents Awarded	Other Research Outputs (please specify)	
No. of outputs arising directly from this research project	1 (submitted, under review) 1 (under preparation)	1			Type	No.

14. Public Access Of Completion Report

(Please specify the information, if any, that cannot be provided for public access and give the reasons.)

Information that Cannot Be Provided for Public Access	Reasons