

RGC Ref. No.: UGC/FDS25/E02/17 _____ (please insert ref. above)
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**RESEARCH GRANTS COUNCIL
COMPETITIVE RESEARCH FUNDING SCHEMES FOR
THE LOCAL SELF-FINANCING DEGREE SECTOR**

FACULTY DEVELOPMENT SCHEME (FDS)

Completion Report
(for completed projects only)

<p><u>Submission Deadlines:</u></p> <ol style="list-style-type: none"> 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.
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Part A: The Project and Investigator(s)

1. Project Title

Study of a low energy input-high water recovery wastewater treatment technology by forward osmosis (FO) using a novel FO draw solution synthesized with hydrophilic and magnetic nanoparticles (MNP)

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

Research Team	Name / Post	Unit / Department / Institution
Principal Investigator	CHAN Cho Yin / Lecturer I	FST/ THEi
Co-Investigator(s)	NA	NA
Others	CHAN Kwan Shing / RA	FST/ THEi
Others	LIU Yan Mei / Final Year Project student	FST/ THEi
Others	WONG Chiu Wai / Final Year Project student	FST/ THEi

3. Project Duration

	Original	Revised	Date of RGC / Institution Approval <i>(must be quoted)</i>
Project Start Date	1/1/2018	NA	NA
Project Completion Date	31/12/2019	30/6/2020	27/5/2019

Project Completion Date	30/6/2020	31/12/2020	18/6/2020
Duration (<i>in month</i>)	24	30	27/5/2019
Duration (<i>in month</i>)	30	36	18/6/2020
Deadline for Submission of Completion Report	31/12/2020	31/12/2021	18/6/2020

Part B: The Final Report

5. Project Objectives

5.1 Objectives as per original application

1. To synthesize a novel magnetic nanoparticles (MNP) with high hydrophilicity for the development of the draw solution that can be applied for forward osmosis (FO) process.
2. To investigate the effects of morphological properties i.e. nanoparticles size distribution, surface charges, specific functional groups attached on the synthesized MNP towards the FO process performance.
3. To evaluate the feasibility of using MNP as FO draw solution for large scale wastewater treatment by using external magnetic field coupled with low energy required membrane technologies.
4. To apply this novel hybrid membranes system for treating with local industrial wastewaters containing complex mixtures.
5. To demonstrate the experimental setup of this proposed study and deliver the concept of zero liquid waste discharge to final year project students, secondary school students and related industries through research & teaching activities as well as laboratory visits and also to further enhance public understanding on how water reuse from wastewater treatment by using an energy efficient and sustainable approach.

5.2 Revised objectives

Date of approval from the RGC: NA

Reasons for the change: NA

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: To synthesize a novel magnetic nanoparticles (MNP) with high hydrophilicity for the development of the draw solution that can be applied for forward osmosis (FO) process.

Magnetic nanoparticles (MNP) (bare Fe_3O_4) was synthesized by using alkaline co-precipitation method. Different types of hydrophilic magnetic nanoparticles were synthesized by using organic ligands such as citrate, polyacrylic acid (PAA), polyethylene glycol (PEG) as well as mesoporous silica layer was attempted in surface coating on the MNP. Among different types of synthesized magnetic draw solution, citrate coated magnetic

nanoparticles conducted at 80°C (80-cit-Fe₃O₄) showed a higher forward osmosis (FO) performance in terms of water flux obtained in the FO process (0.5-0.6 L/m² h).

Objective 2: To investigate the effects of morphological properties i.e. nanoparticles size distribution, surface charges, specific functional groups attached on the synthesized MNP towards the FO process performance.

Morphological properties of both bare iron oxide (Fe₃O₄) and citrate coated magnetic nanoparticles (80-cit-Fe₃O₄) were characterized and critically compared. Results showed that the hydrophilicity of MNP was significantly enhanced after surface coating by citrate at 80°C while the superparamagnetic property of 80-cit-Fe₃O₄ was achieved due to sufficient small nanoparticles size (9-13 nm) and the retained cubic crystalline structure of Fe₃O₄. Besides, the FO performance observed in different pH conditions can also be explained by the charge property of the 80-cit-Fe₃O₄ used as draw solution.

Objective 3: To evaluate the feasibility of using MNP as FO draw solution for large scale wastewater treatment by using external magnetic field coupled with low energy required membrane technologies.

Apart from flat sheet small scale FO membrane was used for optimization study of the FO parameters like flow rate, pH and effect of draw solution concentrations, results showed the FO performance can be scaled up by connecting more of such small unit of FO membrane (i.e. 2-3X of total membrane area) and then more water can be extracted from feed solution under the same treatment time interval. On the other hand, in order to enhance the water quality of the FO collected water, ultra-filtration (UF) and reverse osmosis (RO) were tested as post-treatment step and results showed that RO can achieve a higher salt rejection rate (>99%) than UF within a relatively short processing time i.e. 20 min to treat 3L of FO collected water, thus, a hybrid system of FO/RO can be designed by using this newly synthesized MNP as FO draw solution and the energy consumption is lower when compared to the RO treatment system alone, further scale up of FO experiment to several litre would be possible but it requires a significant longer time for synthesizing the required amount of magnetic nanoparticles and larger area of FO membrane is needed.

Objective 4: To apply this novel hybrid membranes system for treating with local industrial wastewaters containing complex mixtures.

Due to no available sources of real sample can be obtained within this project period that industrial wastewater samples cannot be tested. However, artificial wastewater containing similar pollutants content such as azo dyes (for textile dyeing industry) or inorganic salts (sample assumed with low residual salt concentration after pretreatment) were tested and promising results were observed as water can be extracted from artificial wastewater to the FO draw solution according to their osmotic pressure difference.

Objective 5: To demonstrate the experimental setup of this proposed study and deliver the concept of zero liquid waste discharge to final year project students, secondary school students and related industries through research & teaching activities as well as laboratory visits and also to further enhance public understanding on how water reuse from wastewater treatment by using an energy efficient and sustainable approach.

The experimental setup was demonstrated in different laboratory visits organized for high school and tertiary school students as well as technical staff of different industries. Besides, treatment approach of zero liquid discharge with low energy consumption was discussed in

different teaching courses like Final Year Project (SEV5498/5499), Industrial Wastewater Treatment (SEV5441) and Water and Wastewater Engineering (SEV4221).

5.4 Summary of objectives addressed to date

Objectives <i>(as per 5.1/5.2 above)</i>	Addressed <i>(please tick)</i>	Percentage Achieved <i>(please estimate)</i>
1. To synthesize a novel magnetic nanoparticles (MNP) with high hydrophilicity for the development of the draw solution that can be applied for forward osmosis (FO) process	Yes	100%
2. To investigate the effects of morphological properties i.e. nanoparticles size distribution, surface charges, specific functional groups attached on the synthesized MNP towards the FO process performance.	Yes	100%
3. To evaluate the feasibility of using MNP as FO draw solution for large scale wastewater treatment by using external magnetic field coupled with low energy required membrane technologies.	Yes	90%
4. To apply this novel hybrid membranes system for treating with local industrial wastewaters containing complex mixtures.	Yes	80%
5. To demonstrate the experimental setup of this proposed study and deliver the concept of zero liquid waste discharge to final year project students, secondary school students and related industries through research & teaching activities as well as laboratory visits and also to further enhance public understanding on how water reuse from wastewater treatment by using an energy efficient and sustainable approach.	Yes	100%

6. Research Outcome

6.1 Major findings and research outcome

(Maximum 1 page; please make reference to Part C where necessary)

Bare magnetic nanoparticles (Fe_3O_4) were obtained by alkaline co-precipitation method. A higher FO performance was observed in citrate-coated magnetic nanoparticles synthesized at 80°C (80-cit- Fe_3O_4) and used as draw solution of the FO process, on the other hand, surface coating conducted at room temperature as well as by using other ligands (i.e. PAA, PEG polymers) resulted in lower FO performance so that 80-cit- Fe_3O_4 was selected for further investigation on the FO process and material characterization. Results showed that the synthesized 80-cit- Fe_3O_4 has 2.89 wt% of carbon and 0.40 mmol citrate loading (/g of 80-cit- Fe_3O_4). XRD result proved the face-centered cubic structure of magnetite Fe_3O_4 , and retained the nanoparticles' crystalline structure without showing significant change after coating of citrate. Both asymmetric and symmetric stretchings of COO- group of citrate were verified by the band at 1620 cm^{-1} and 1380 cm^{-1} shown in FTIR pattern. TEM image indicated that the 80-cit- Fe_3O_4 was observed in spherical to slightly irregular shape with diameter of 9-13 nm. VSM showed that the saturation magnetization of 80-cit- Fe_3O_4 was 82.6 emu/g, thus exhibited superparamagnetic property and simple regeneration of draw solution can be performed by applying external magnetic field. Otherwise, high energy consumption method like RO is required to collect and concentrate this draw solution after the FO process.

FO performances of 80-cit- Fe_3O_4 was observed lower than sodium citrate solution (reference). Although higher osmotic potential of sodium citrate solution can extract more water from the feed solution, its regeneration by using RO requires higher energy consumption, moreover, membrane clogging and leakage of this low molecular mass draw solutes to feed solution greatly affected the FO performance. Results showed that 0.3 LMH ($\text{L}/\text{m}^2\text{h}$) was observed in 150 g/L of 80-cit- Fe_3O_4 when used as draw solution of the FO process at pH 6 with 290 mL/min of flow rate. Higher concentration such as 350 g/L of 80-cit- Fe_3O_4 showed higher performance in water extraction (i.e. 0.54 LMH) due to higher osmotic pressure difference exerted between two solution sides. Besides, neutral to slightly alkaline pH would be more favorable to the FO process because lower pH of draw solution (i.e. pH 4) increased positive charge particles attraction on the negatively charged FO membrane surface resulting in lower water extraction. At higher pH (i.e. pH 9-10) due to increased deprotonation of the tricarboxylic acid groups thus more stable dispersion of the 80-cit- Fe_3O_4 facilitated the FO process, in addition, more negatively charged of draw solutes at higher pH increased the electrostatic repulsion within the FO membrane that can prevent membrane fouling and thus a higher water flux can be achieved. On the other hand, results indicated that the water extraction rate was enhanced from 0.26 to 0.48 LMH when the flow rate of FO process increased from 162 mL/min to 290 mL/min, as higher flow rate of FO operation can maintain high osmotic pressure difference between both solution sides resulting in higher water flux.

Artificial wastewater samples containing 30 mg/L of methylene blue or 10mM NaCl low salt solution were treated separately by the FO process using 250 g/L 80-cit- Fe_3O_4 as the draw solution. Two FO cell units were connected to increase the treatment capacity and the amount of FO treated water for RO post-treatment. Results showed that 0.16-0.25 LMH water fluxes were achieved and such reduced water extraction rates in both artificial wastewater samples comparing to deionized water was due to the reduced osmotic pressure between the two solution sides resulting in lower water flux. Post-treatment of this FO treated water was conducted by using RO and results showed that high quality of FO/RO treated water can be obtained for water reuse. Besides, magnetic separation of 80-cit- Fe_3O_4 was performed using a magnet (0.5 T). After regeneration, 80-cit- Fe_3O_4 was reused as the draw solution and no significant drop in water flux was observed (<5% difference) when compared to the first FO cycle indicating that the synthesized magnetic nanoparticles were functionally stable. Furthermore, results showed that both salt and dye rejection rates were very high (>99.9%) and the reversed solute fluxes observed in 0.17-0.23 $\text{g}/\text{m}^2\text{h}$ which were significantly

lower than the values reported in past studies using small inorganic salt as the draw solution. As a result, the problem of solutes clogging on FO membrane surface can be reduced and the FO membrane lifespan can be prolonged for further treatment. Moreover, this proposed FO/RO treatment system is more energy efficient than using RO alone for the wastewater treatment.

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

The FO performance of synthesized magnetic nanoparticles used as draw solution should be further evaluated by using different types of FO membranes. Since past studies reported that different materials used in fabrication (i.e. cellulose triacetate and thin-film composite membrane) and membrane configurations (i.e. hollow fiber, spiral wound compared with flat sheet membrane) may significantly affect the properties of membrane permeability, membrane fouling and concentration polarization that eventually may affect the FO performance. Besides, apart from the concerns of energy usage and water recovery, techno-economic analysis should also be performed to compare the results of current study with different types of FO draw solution used as well as different integrated treatment systems selected.

7. Layman's Summary

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

As increasing demand of wastewater treatment is required due to rapid population growth, a lot of energy (2-3% of global uses) is consumed resulting in high carbon emission and accelerated global warming. In order to reduce such carbon emission for environmental protection, a new wastewater treatment approach with low-energy input and high-water recovery is investigated by using forward osmosis (FO). FO can be operated under relative low pressure and thus less energy is used when comparing to the conventional reverse osmosis (RO) process. An appropriate working draw solution (DS) is required to provide a constant driving force along the FO process to extract water from wastewater feed stream. In this study, a newly synthesized magnetic nanoparticles bearing both hydrophilic and magnetic properties is used as an effective FO draw solution, the diluted DS after the FO process can be easily collected and reused after simple magnetic separation by a magnet. On the hand other, high quality of FO treated water can be collected for simple and rapid RO post-treatment to enhance the water quality, thus the energy use for treating same amount of wastewater can be reduced and also high water recovery is achieved by using this FO/RO hybrid system.

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)						
2022 (to be published)	2022 (to be accepted)	2021 (under peer review)	NA	Kwan-Shing Chan, Dawson Wai-Shun Suen, Chi-Wing Tsang, Ka-Wing Wong, Yan-Mei Liu, Chiu-Wai Wong, Cho-Yin Chan *	Wastewater treatment and water recovery by forward osmosis (FO) using magnetic nanoparticles as draw solution submitted in Journal of Visualized Experiments	No	Yes (manuscript)	Yes	To be updated

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)
March 2021, Singapore	Wastewater treatment and water recovery by forward osmosis (FO) using magnetic nanoparticles as draw solution	The 5th ICSTR Singapore – International Conference on Science & Technology Research, Scientific & Technical Research Association (STRA). Singapore. 26-27, March 2021 (Oral online presentation).	No	Yes	Yes	To be updated

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

(Please elaborate)

- Selected experiments of FO process and parameters optimization have been arranged for training 2 Final Year Project students (SEV5498/5499)
- The ideas and treatment approach have been discussed in modules SEV5441 Industrial Wastewater Treatment and SEV4221 Water and Wastewater Engineering for showing how to achieve energy saving in wastewater treatment
- A poster showcase has been arranged in laboratory to share the research ideas with students and visitors

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
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]
[REDACTED]	[REDACTED]	[REDACTED]	[REDACTED]

12. Other Impact

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

Based on this project finding, PI has been invited as a guest editor of a special issue “Magnetically separable nanoparticle and single-atom catalysts for environment and energy applications” in Journal of Visualized Experiments (JOVE)

13. Statistics on Research Outputs

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

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
NA	NA