# Techno-economic Assessment of Scale-up of Bio-flocculant Extraction and Production by using Okra as Biomass Feedstock

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## Abstract

This paper reports a techno-economic assessment for industrial scale bio-flocculant production with okra as biomass feedstock. The sludge dewatering ability of the bio-flocculant was evaluated prior to economic analysis. Several optimisation strategies were investigated in order to lower the bio-flocculant production cost. The results showed that continuous mode microwave extraction was more economically beneficial than conventional extraction in batch and continuous modes. Sensitivity analysis revealed that the production cost was significantly affected by annual production and extract yield, and moderately influenced by raw material price. The optimised scheme for bio-flocculant production was continuous mode microwave extraction at 90˚C, a residence time of 10 minutes, a water loading of 3.5 w/w and production rate of 220 tonnes per year. The economic assessment showed that the gross margin was positive, return on investment was in the expected range of 20 to 30% and payback time was within 5 years.

Keywords: extraction, okra bio-flocculant, sludge dewatering, SuperPro Designer, economic analysis, scale-up

| **Abbreviations** | **Full Words** |
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| | |
|---|---|
| AF | aqueous bio-flocculant |
| BPE | boiling point elevation |
| C- | conventionally extracted |
| CSE | conventional solvent extraction |
| DFC | direct fixed capital |
| DF | dried bio-flocculant |
| EPC | equipment purchase cost |
| IRR | internal rate of return |
| M- | microwave extracted |
| MAE | microwave assisted extraction |
| NPV | net present value |
| PBT | payback time |
| ROI | return on investment |
| SS | suspended solids |

## 1. Introduction

The use of polymeric flocculants particularly polyacrylamides is debatable because their application may cause environmental consequences and health hazards. Contamination of water and wastewater treatment with residual unreacted acrylamide monomer after application of polyacrylamide has been reported (Hesami, 2014). Acrylamide is identified as a suspected carcinogen and extremely toxic causing severe neurotoxic effects (Besaratinia and Pfeifer, 2007). Moreover, polyacrylamide biodegradation is slow (Bolto, 2007; Brostow, 2009) and the release of acrylamide could enter the food chain and cause carcinogenic effects (Sharma, 2006). With increasing awareness of the potential harm caused by polyacrylamides, plant-based bio-flocculants have been studied in the past 10 years (Lee, 2014) with the aim to replace polyacrylamides.

Conventional solvent extraction (CSE) in water bath coupled with heat and agitation is commonly employed to extract the bio-flocculants from plant materials with polysaccharides as the main components by using water as solvent (Lee, 2014). In our previous study (Lee, 2015), a more efficient CSE method with shorter extraction time than previously published methods was designed to extract okra bio-flocculant for application in sludge dewatering. Nevertheless, the extraction performance was unsatisfactory because bio-flocculant with high dewatering properties was best extracted at temperatures ≥70 ˚C, but a reduction in yield was observed due to the long extraction time of 2 hours.

Numerous workers have investigated the use of microwave assisted extraction (MAE) to extract polysaccharides from plant materials such as orange peel (Yeoh, 2008), Pressed Berry Residue (Katalin, 2012) and seaweed (Rodriguez-Jasso, 2011). These studies demonstrated that microwave extraction has the advantages of higher yield and shorter extraction time than conventional extraction. In order to further reduce the extraction time, increase the extraction yield and enhance the bio-flocculant quality; MAE was employed to extract okra bio-flocculant. Our recent publication (Lee, 2016) has demonstrated that MAE presented a different mechanism when compared to CSE method. Microwave selective heating of okra was found to occur at extraction temperatures above 50 ˚C and this led to significantly higher yield (49% vs 26%) than CSE, with a short extraction time of 10 minutes and high temperature ≥70 ˚C.

In this paper, the sludge dewatering abilities of microwave extracted bio-flocculants are reported and compared with conventional extracted bio-flocculants and commercial flocculants (polyacrylamides) at various dosages. The dewatering activities were evaluated with reference to suspended solids (SS) removal after filtration and reduction of sludge volume which was represented by water recovery after dewatering process. Water recovery is inversely proportional to sludge volume; which means that higher water recovery denotes lower sludge volume. These results are highly important for application of bio-flocculants in sludge dewatering because it indicates the quality of flocs formed (size and density) and the dewaterability of the sludge.

The results of an economic feasibility study to check the viability of producing bio-flocculant with conventional and microwave extractions at industrial scale are then presented in order to compare the performance of okra-derived bio-flocculants with polyacrylamides in terms of quality and cost. Microwave heating has been shown to lead to wider process engineering benefits when carried out at scale, such as a reduction in equipment size and simplification of processing steps when compared to conventional methods, which results in further economic benefits (Robinson, 2010). However, the evaluation of the economic analysis of microwave extraction of natural bio-products has not been reported previously. Taking new processes to industry requires not only technical feasibility, but also economic attractiveness in relation to product price and production cost. In this work, the economic feasibility of scaling up the bio-flocculant production to demonstration scale was investigated via the use of SuperPro Designer v9.0 simulation software. The design of a demonstration scale plant (semi-works plant) will closely resemble that of the anticipated future commercial/full scale plant with typical reactor volumes in the 100 - 1000 litre range (Liu et al., 2016).

The main objective is to model and design an economically viable bio-flocculant production process with low production cost by using okra as the biomass feedstock. In order to lower the production cost at large scale production, several optimisation strategies were investigated including (i) batch versus continuous modes, (ii) microwave versus conventional extraction processes, (iii) investigation of the effects of annual production, extraction yield and raw material okra price on the production cost through sensitivity analysis. Finally, the economic performance of the optimised production scheme was justified and its estimated selling price was compared with polyacrylamides and food grade bio-flocculants.

## 2. Materials and Methods

### 2.1 Experimental microwave extraction of bio-flocculant and sludge dewatering analysis

The cationic (FO 4400 SH) and anionic (AN 934 SH) polyacrylamides were obtained from SNF Floerger. The steps in conventional (CSE) (Lee, 2015) and microwave (MAE) (Lee, 2016) extractions of bio-flocculant and the examination of their dewatering abilities were reported in our previous publications.

### 2.2 Economic feasibility study

The simulation includes a mathematical model that performs material and energy balance calculations for every operation within the equipment. An example of material balance and the results of energy balance were presented in Tables S1 and S2 in supplementary material. The lab scale optimised extraction and operating conditions are used to simulate CHE and MAE processes, and the design specification of equipment was set by referring to literature and design handbooks. The basis of the full scale process assumes that the process will have identical performance with respect to the extraction yield as observed at laboratory scale if the same conditions are used.

The process plant is designed to be based in the United Kingdom due to the rapid growth of microwave technology for industrial applications in U.K. and the U.K. regulations are followed in the whole design process. The annual operating time is fixed at 8000 hours (Heinzle, 2006) and the operating lifetime is assumed as 20 years. Not all the components registered in the design process are available in the software component database, and hence are approximated using reference components with similar physical/chemical properties (Table 1).

Table 1: All the components used in simulation flowsheet

| **Components** | Nitrogen | Oxygen | Water | Fresh okra | Okra pods | Okra seeds | Okra extract | Extract waste |
|---|---|---|---|---|---|---|---|---|

| **Reference component** | Nitrogen | Oxygen | Water | Biomass | Biomass | Biomass | Cellulose | Biomass |
|---|---|---|---|---|---|---|---|---|
| **Databank source** | Designer | Designer | Designer | Designer | Designer | Designer | User-defined | Designer |

### *2.2.1 Process description*

The bio-flocculant production process is proposed as: vegetable washer → vegetable seed remover → vegetable slicer → conventional or microwave extractor → plate & frame filter (batch process) or hydrocyclone (continuous process) → forced circulation evaporator → rotary dryer. An example of the process flowsheet is illustrated in Figure 1.

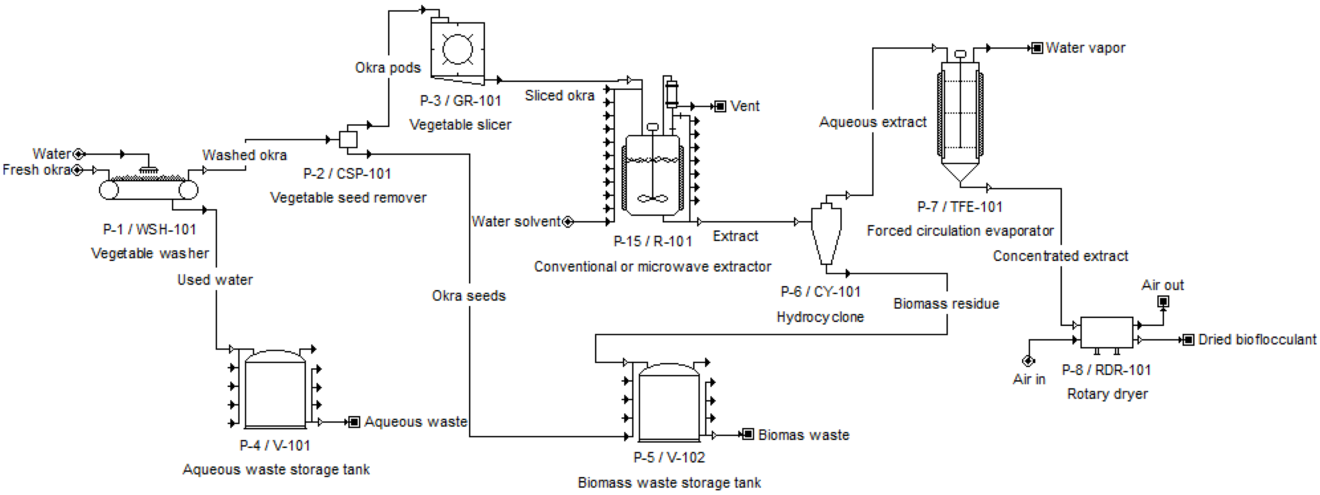

Figure 1: Process flowsheet of bio-flocculant production

The supply of fresh okra is firstly washed with water in a vegetable washer. Afterwards, the seeds are separated from the okra pods with a vegetable seed remover. The okra pods are then sent to a vegetable slicer to produce sliced okra with approximate width value of 0.1cm. The sliced okra is then transferred to an extractor (conventional or microwave) for heterogeneous solid (sliced okra) and liquid (water solvent) extraction to obtain okra extract. For the conventional extractor a turbine impeller was chosen as the mechanical agitator because it is excellent for providing circulation and effective for fluids with high viscosity (Coulson, 1999). The calculations of the agitation speed and the required power are presented in the supplementary material (section S.1). For microwave extraction, a microwave generator is required to generate electromagnetic energy which is delivered by a transmission line to the microwave extractor for heating the process materials.

The next step is the separation of aqueous extract from the biomass residue with a plate & frame filter (batch operation) or hydrocyclone (continuous operation) with the assumption that 5% of the aqueous extract is lost and discharged as cake with the extract residue. The aqueous extract could be collected at this point and sold as aqueous bio-flocculant (AF). The calculation of the filtration period is explained in the supplementary material (section S.3).

A forced circulation evaporator is used to remove as much water as possible (50-70%) to concentrate the product prior to drying. This evaporator is the most commonly used within the food industry and suitable for processing viscous and heat-sensitive materials (Fellows, 2009). It is operated at low pressure (50 mm Hg) to reduce the partial pressure of water within the gas phase and promote water removal at a temperature of 50 $^{o}$C (Chung, 2012), thus minimising decomposition or alteration of heat-sensitive materials. The typical heat transfer coefficient of 3kW/m$^2$.K is assumed for condensing steam, which is a common heating medium (Seader, 2011). Boiling point elevation (BPE) is assumed to be negligible in heat transfer calculations because the high molecular weight of pectin-like polysaccharides (~$10^6$ Da (Heiwall, 2007)) extracted from okra pods gives negligible BPE (Saravacos, 2011).

Finally, the concentrated bio-flocculant is dried in a rotary dryer to get dried bio-flocculant (DF). This dryer is suitable for large scale drying of heat-sensitive materials by using hot air as the drying medium. When the hot gases that are flowing through the rotating cylinder come into immediate contact with the wet material, the gas temperature is lowered rapidly and the material is heated and its moisture content is reduced (Richardson, 2002). The evaporative capacity is assumed as 28.8 kg.m$^3$/h (Richardson, 2002) and the power consumption is estimated as 0.16 kW/m$^2$ (Green, 2007). The calculation of the drying time is explained within the supplementary material (section S.2).

### *2.2.2 Economic analysis*

Prior to economic evaluation, various inlet and outlet streams of the process were classified as raw material, revenue, waste or emission and then specified with their associated economic values. The preliminary economic evaluation for manufacturing a bio-product usually involves the estimation of capital investment, operating costs and profitability analysis.

#### *2.2.2.1 Capital cost estimation*

The total capital investment is made up of direct fixed capital (DFC), working capital and start-up and validation cost. The DFC is the total cost of the process ready for start-up and not recovered at the end of the project life (Towler, 2013). Various items of DFC as listed in Table 2 are estimated based on the total equipment purchase cost (EPC) using several multipliers sometimes called "Lang Factors" (Harrison, 2015).

Table 2: Estimated values of multipliers for various items of DFC (Harrison, 2015)

| Cost item | Average multiplier |
|---|---|
| ***Total process direct cost (TPDC)*** | |
| Equipment purchase cost (EPC) | |
| Process piping | 0.40 x EPC |
| Instrumentation | 0.35 x EPC |
| Insulation | 0.03 x EPC |
| Electrical facilities | 0.15 x EPC |
| Buildings | 0.45 x EPC |
| Yard improvement | 0.15 x EPC |
| Auxiliary facilities | 0.50 x EPC |
| Unlisted equipment installation cost | 0.5 x unlisted equipment purchase cost |
| ***Total process indirect cost (TPIC)*** | |
| Engineering | 0.25 x TPDC |
| Construction | 0.35 x TPDC |
| ***Total process cost (TPC)*** | TPDC + TPIC |
| Contractor’s fee | 0.05 x TPC |
| Contingency | 0.10 x TPC |
| ***Direct fixed capital (DFC)*** | TPC + contractor’s fee and contingency |

The EPC is the sum of the listed equipment purchase cost and the unlisted equipment purchase cost (Heinzle, 2006). The unlisted equipment (e.g. storage silo for okra raw material, tank for storage of water from main supply, pumps, valves, transportation conveyor, condenser and vacuum pump for evaporator and so on) purchase cost is assumed to be 20% of the major equipment cost (Athimulam, 2006). The equipment purchase cost was obtained from equipment vendors and published cost data (Couper, 2012; Towler, 2013) and was updated to March 2016 with Chemical Engineering Plant Cost Index = 556.8 (Jenkins, 2016; Leland Blank 2011).

The materials of construction with characteristics of corrosion resistance, robustness and weatherable are selected for each equipment. The material cost factors for different materials of construction for prices relative to carbon steel (Sinnott, 2005; Towler, 2013) are considered in the calculation of EPC. The installed cost of each item of equipment is also considered in the simulation and calculated by multiplying the purchase price with installation multiplier (Couper, 2012). These results are summarised in Table S1 within the supplementary material.

Working capital is needed to start the process up and operate it until income is earned and is usually in the range of 10 to 20% of DFC (Silla, 2003). In this work, the expenses of working capital comprise of raw materials and labour and utilities for 4 months, negligible waste treatment and other miscellaneous expenses (Heinzle, 2006), which gives the amount of 18% of DFC. Start-up and validation costs are the costs of making the transition from construction to operation (Neil, 2005) and a common value of 20% of DFC is included in simulation (Harrison, 2015).

### *2.2.2.2 Operating cost estimation*

The operating cost to run a process is the sum of all on-going expenses and can be divided into variable and fixed costs as summarised in Table 3.

Table 3: Estimated value for various components of operating cost

| **Components of operating cost** | **Estimated value** |
|---|---|
| ***Variable costs (Pereira, 2013)*** | |
| 1. Raw materials | 1 \$/kg for fresh okra (21 June 2016), 1.1 \$/m$^3$ for water (2015/2016) |
| 2. Utilities | 0.1 \$/kW.h for electricity (2016b), 12 \$/MT for steam (Harrison, 2015), 0.13 \$/kW.h for generation of microwave energy |
| 3. Operating labour | 30\$/hour |
| 4. Waste treatment | Negligible |
| 5. Shipping and packaging | Negligible |
| ***Fixed costs (16 March 2016; Harrison, 2015; Sinnott, 2005)*** | |
| 1. Maintenance of labour and materials (including equipment spares) | 5% of DFC |
| 2. Depreciation of fixed assets | 10% of DFC |
| 3. Laboratory, quality control and quality assurance cost | 20% of operating labour |
| 4. Supervision | 20% of operating labour |
| 5. Insurance | 1% of DFC |
| 6. Local (property) taxes | 2% of DFC |
| 7. Factory expenses (e.g., accounting, payroll, security and cafeteria) | 5% of DFC |
| 8. Miscellaneous expenses such as R&D and other | Negligible |

Variable costs are the expenses that vary with the production rate, contrary to fixed costs. It increases when the production rate is high and decreases with low production (Pereira, 2013). The current wholesale price of okra at \$1/kg was obtained from a trading supplier in 2016 (21 June 2016). Utility cost was estimated based on mass and energy balance from the simulation which determined the quantities of each type of utility required (Lam, 2014). Electricity is used to run the equipment and generate microwave energy. As 30% of the generated microwave energy will be consumed by the auxiliary equipment associated with the microwave unit, hence 143 kWh of electricity is required to get 100 kWh of microwave energy used for heating. Steam is used as the heating medium in the evaporator and conventional extractor. It is assumed that the steam is available and purchased for direct application.

The number of operators per shift for each unit in batch and continuous processes is shown in Table 4, while an example of estimation of operator requirement is summarised in Table 5 below.

Table 4: Typical operator requirement for each equipment (Green, 2007)

| | **Batch process** | **Continuous process** |
|---|---|---|
| **Operators per unit per shift** | Vegetable washer: 0.1 | Vegetable washer: 0.1 |
| | Seed removal: 0.1 | Seed removal: 0.1 |
| | Vegetable grinder: 0.1 | Vegetable grinder: 0.1 |
| | Batch extractor: 1 | Continuous extractor: 0.5 |
| | Plate & frame filter: 1 | Hydrocyclone: 0.25 |
| | Evaporator: 0.25 | Evaporator: 0.25 |
| | Rotary dryer: 0.5 | Rotary dryer: 0.5 |
| | Aqueous waste storage tank: 0.2 | Aqueous waste storage tank: 0.2 |
| | Solid waste storage tank: 0.2 | Solid waste storage tank: 0.2 |
| | Auxiliary equipment: 0.2 | Auxiliary equipment: 0.2 |
| **Total operators per shift** | 3.65 rounded up to 4 | 2.4 rounded up to 3 |

Table 5: Estimation of operator requirement (Green, 2007)

| | **Subject** | **Calculated results** | **Remarks** |
|---|---|---|---|
| **General** | Annual operating hours | 8000 | 48 weeks in a year |
| | Annual working hours / operator | 1795 | Average hours worked by an operator is 37.4 weekly (Thomas, 2016) |
| | Annual labour cost ($) / operator | 53856 | Labour cost is $30 per hour. |
| | Number of operators / year | 4.5 rounded up to 5 | Assumption: 1 operator per shift |
| **Batch process** | Number of operators / year | 20 | 4 operators per shift |
| | Annual operator cost ($) | 1077120 | 20*53856 |
| **Continuous process** | Number of operators / year | 15 | 3 operators per shift |
| | Annual operator cost ($) | 807840 | 15*53856 |

As shown in Table 5, the operator cost is higher for batch process due to the lower level of automation. Continuous processes employ automated systems, which implies fewer operators and lower operator cost (Pereira, 2013). In this specific case the continuous extractor and hydrocyclone have a lower operator demand than the corresponding batch extractor and filter system, resulting in a lower overall operator requirement for the continuous process.

Aqueous waste is generated from the vegetable washer and it may contain some soil and microorganism. It is treated and reused for washing of okra and the treatment cost is redeemed through savings in water cost. The solid waste generated consists of okra seeds and wet biomass residue, which are essentially organic materials and biodegradable. It can be sold or given to the agricultural production sector to be used as bio-fertiliser (Wang, 2007) or applied in anaerobic digestion to produce biogas (22 January 2013). Okra seeds could be used for production of edible oil and okra seed flour (Kumar, 2010). For these reasons, the waste treatment cost was taken to be negligible (Pereira, 2013). The wastes generated are stored with the base assumption of at least 24 hours in thermoplastic tanks prior to transportation to another site for reuse or for other application.

#### *2.2.2.3 Profitability analysis*

The revenues are from sales of the main products, which is either AF or DF. Dividing the annual operating cost by the annual production rate yields the unit production cost of bio-flocculant ($/kg) (Harrison, 2015). The economic measures to assess the profitability include gross margin, return on investment (ROI), payback time (PBT), internal rate of return (IRR) and net present value (NPV) (Harrison, 2015).

The sum of product revenues minus the raw material costs is known as gross margin (Towler, 2013) and represents the proportion of each dollar of revenue that the company retains as gross profit (Vučurović, 2012). The ROI is defined as annual operating income after tax divided by total capital cost and 20 to 30% is expected (Athimulam, 2006; Sinnott, 2005). ROI measures how effectively the company uses its invested capital to generate profit (Heinzle, 2006). The PBT is the time required to pay off the initial investment from income (Sinnott, 2005) and estimated by dividing the total capital cost by the average annual income where the taxes and depreciation are neglected (Towler, 2013). Typically, a PBT of 2 to 5 years is expected (Sinnott, 2005).

NPV is the sum of the present values of the future cash flows (Heinzle, 2006). The IRR is a discount interest rate that makes the NPV of all cash flows equal to zero. It is a measure of the maximum interest rate that the project could break even by the end of the project life (Towler, 2013). If the NPV is positive by assuming a IRR of 7%, it indicates that the projected earnings exceed the anticipated costs (all in present dollars) and this investment will be profitable (Vučurović, 2012).

## **<u>3. Results and Discussion</u>**

### **3.1 Justification of sludge dewatering performance of bio-flocculants**

The dewatering abilities of microwave extracted bio-flocculants were evaluated through comparison with conventionally extracted bio-flocculants (results were taken from previous publication (Lee, 2015)) and polyacrylamides at various dosages, and the results are displayed in Figure 2.

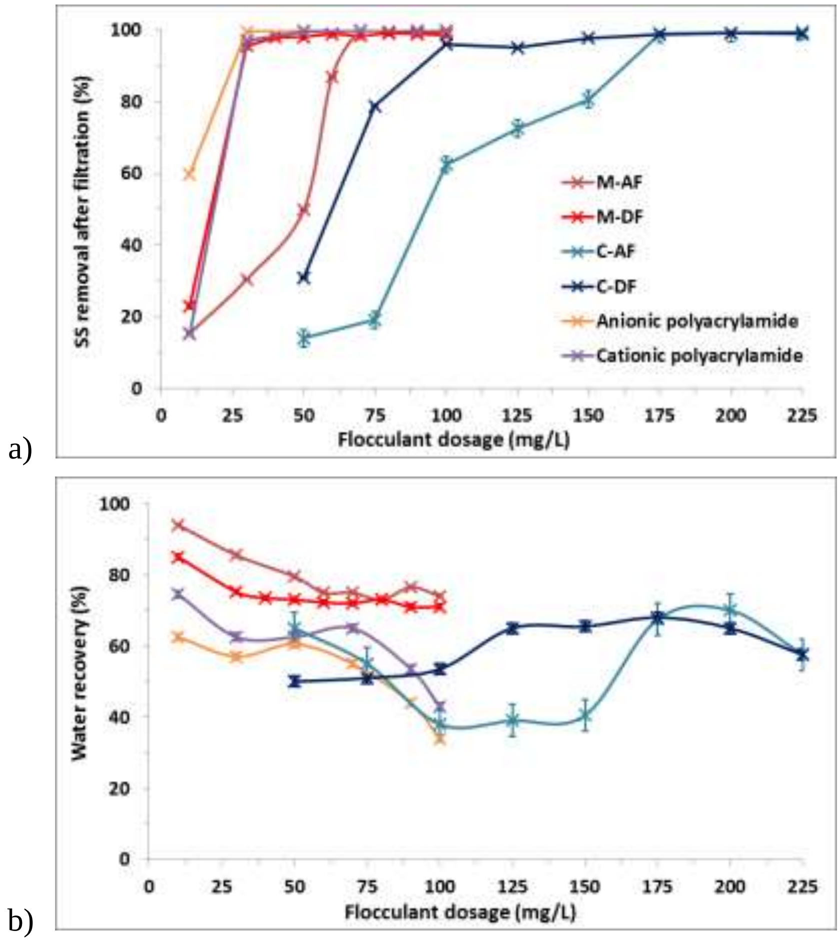

Figure 2: Comparison of dewatering abilities of different flocculants in terms of (a) SS removal after filtration and (b) water recovery (Notes: C-: conventionally extracted, M-: microwave extracted, AF: aqueous bio-flocculant, DF: dried bio-flocculant)

As shown in Figure 2, microwave-extracted dried bio-flocculants (M-DF) require a smaller optimum dose (30 mg/L) to attain the highest SS removal (≥99%) and water recovery (≥75%), however conventionally extracted dried bio-flocculants (C-DF) required four times the dosage at 125 mg/L in order to achieve comparable performance. It is likely that microwave extraction enhances the release, diffusion and solubility of high molecular weight biopolymers (polysaccharides) with dewatering properties into the solvent, and accelerates the mass transfer of biopolymers from the interior of plant cell to the solvent phase (Wei, 2010).

The results also showed that M-DF achieved the optimum SS removal after filtration (≥99%) at the same dosage (30 mg/L) as anionic and cationic polyacrylamides. However, M-AF required a higher dosage at 70 mg/L to achieve similar SS removal efficiency as polyacrylamides. The dewatering activities of AF were examined on the second day after extraction and due to high water content (>80%) in the extract; hence the active constituents with dewatering properties are more susceptible to microbial attack and biodegradation may subsequently occur during the storage time (León-Martínez, 2011). Conversely, fresh extracted AF were sent for drying to obtain DF and it has been reported that removal of moisture through the low temperature drying process (<60˚C) could minimise the degradation that occurs with time in the aqueous phase and thus preserve the active ingredients in the DF (Harbourne, 2009; Pirbalouti, 2013).

In addition, M-AF and M-DF displayed higher water recoveries than polyacrylamides at all dosages, which denotes that lower sludge volume is achieved by using bio-flocculants. This is a great advantage of bio-flocculant because reduction in sludge volume facilitates the sludge handling and transportation and most importantly decreases the sludge management cost. These results indicate that M-DF exhibits comparable or even higher dewatering performance than polyacrylamides.

## 3.2 Economic analysis of bio-flocculant production

### *3.2.1 Batch versus continuous operations*

The batch extractions with microwave method are restricted to smaller scale with annual production less than 50 tonne/year due to limitation of maximum power (100 kW) that could be generated by the industrial microwave generator. Due to this reason, the comparative study between batch and continuous processes for conventional and microwave extractions was conducted up to 50 tonne/year of annual production and the results are presented in Figure 3.

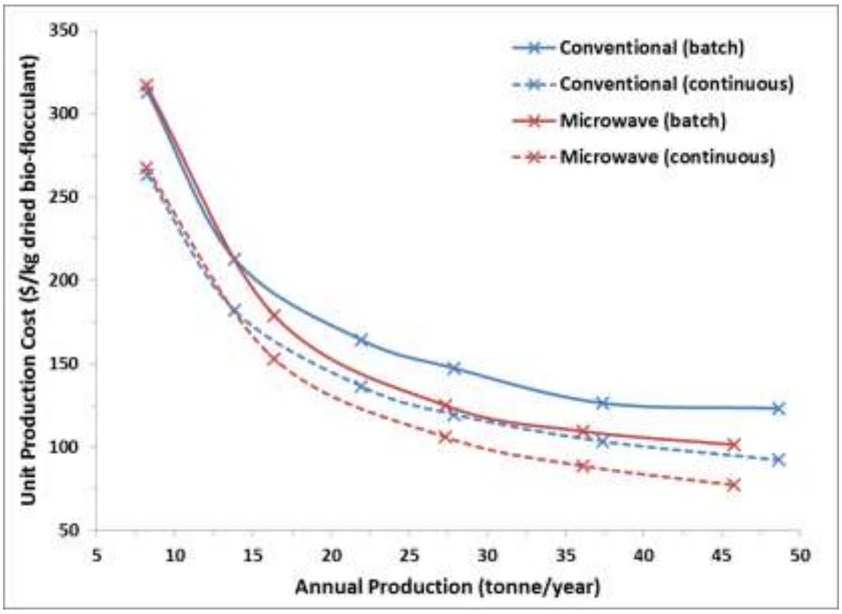

Figure 3: Batch versus continuous operations of conventional and microwave extraction of bio-flocculants

The results show that the unit production cost for continuous operation was significantly lower than batch operation for both conventional and microwave extraction processes. It indicates that continuous processes that operate throughout the year tend to be more economically beneficial and profitable. The significant cost components that contributed to the difference in production costs between batch and continuous microwave processes on the basis of similar annual production at 45.8 tonne/year are shown in Figure 4.

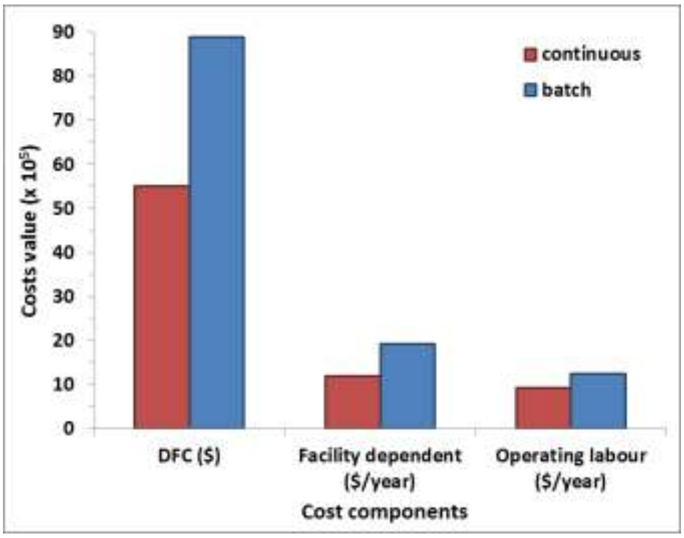

Figure 4: Comparison of DFC, facility dependent and labour costs for batch and continuous microwave processes

For production at 45.8 tonne/year for instance, the flow rate of okra feedstock is 700kg per batch and 170 kg/h for continuous microwave process. It is clear that batch processes require larger equipment for processing of larger amounts of material, which directly increases the equipment cost and then contributes to the increase in DFC. Consequently, facility dependent costs which comprise of maintenance, depreciation, taxes, factory expenses and insurance were raised because these costs are directly influenced by DFC.

As presented in Figure 4, the DFC and facility dependent costs of batch process are 38% higher than continuous process. Furthermore, the operating labour cost for the continuous process was 25% less than the batch process because the operation of the continuous process is fully automated, which reduces the operator requirement. All these results revealed that the continuous process exhibits the advantage of lowering the production cost and hence, further study was conducted on the basis of continuous mode.

### *3.2.2 Conventional versus microwave extractions*

The economic feasibility of conventional versus microwave extractions of bio-flocculants on a continuous basis is compared by observing the effect of increasing annual production on the unit production cost and the results are displayed in Figure 5.

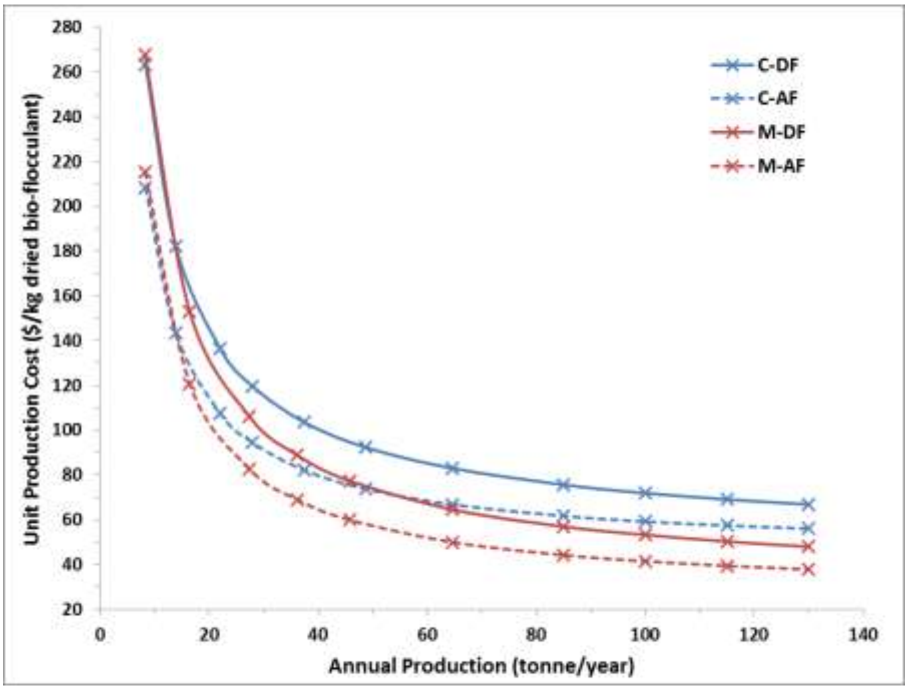

Figure 5: Conventional versus microwave extractions of bio-flocculants

As shown in Figure 5, microwave processing showed similar unit production cost as conventional extraction for small scale production less than 15 tonne/year due to the high capital investment of the microwave generator. Conversely, for larger production capacity, microwave extraction displayed its economic attractiveness as the unit production cost was notably lower than the conventional process. Based on similar production rate (for instance

64.6 tonne/year, Table 6) for conventional and microwave extractions, microwave extraction requires less feedstock, smaller equipment for processing and extraction and a lower heating duty due to a higher extraction yield and shorter extraction time than conventional extraction. All these factors contributed to the lower operating cost for MAE when compared to CSE. The capital cost for MAE to produce 64.6 tonnes/year was similar to the CSE process due to the large capital investment for the microwave generator, however the lower operating cost for MAE leads to a significant reduction in the unit production cost. A further reduction in the unit production cost for MAE is expected due to the learning curve effect with this new technology, which will result in a capital cost reduction for subsequent process units (Levente, 2017).

Table 6: Comparison of conventional and microwave extraction of bio-flocculants on continuous mode at annual production of 64.6 tonne/year

| **Comparison parameter** | **CSE** | **MAE** |
|---|---|---|
| Okra feeding rate to vegetable washer (kg/h) | 415.2 | 240.5 |
| Extraction conditions | 70 °C, 2 hours, 3.5 w/w water loading | 70 °C, 10 mins, 3.5 w/w water loading |
| Heating duty for extraction (kW) | 58.19 | 45.2 |
| Extractor volume (L) | 2558.3 | 159.3 |
| Mass coefficient for extract yield | 0.0256 | 0.0442 |
| Production capital cost x $10^6$ (\$) | 7.98 | 7.63 |
| Production operating cost x $10^6$ (\$/year) | 5.37 | 4.18 |
| Unit production cost (\$/kg DF) | 83.04 | 64.6 |

In addition, Figure 5 also shows that the production costs of AF for both extraction processes were 15 to 25% less than DF due to the elimination of investment cost in evaporation and drying steps. For the case in water treatment whereby the flocculation and sludge dewatering are the common process, investment in an onsite production process of AF is possibly viable. The extracted AF is transferred to nearby treatment sites and applied directly to minimise the risk of degradation of bio-flocculant quality. Even though initial investment cost is required for construction of onsite AF process, but this amount could be counterbalanced from long term perspective through significant saving from purchasing DF.

### *3.2.3 Effects of annual production, extract yield and okra price on the unit production cost*

#### *3.2.3.1 Effect of extract yield*

The following discussion is about the effects of extract yields and raw material okra price to the unit production cost on the basis of 100 tonne/year of annual production in order to explore more possible scenarios to lower the production cost. Each parameter was varied individually while all other variables were kept constant. The impacts of extract yields with respect to variations of extraction temperature and time and solvent loading to the unit production cost were investigated because those extraction parameters showed significant effects on yields in our previous publication (Lee, 2016).

By referring to the operating cost breakdown shown in Figure 6 below, the costs of the total raw materials are the dominant factor that manipulates the overall operating cost. The okra cost is the largest contributor to the raw material cost due to low amount of desired compounds in the plant material. The okra cost can fluctuate markedly depending upon the weather condition, supply capacity and the market demand. Hence, the influence of okra cost on the production cost is also discussed in this section.

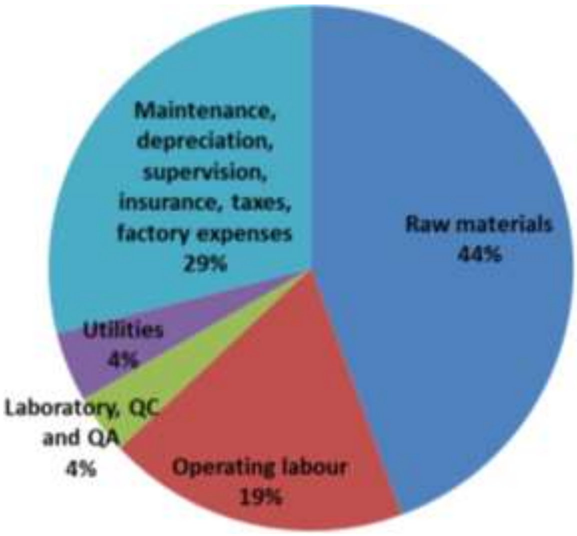

Figure 6: Operating cost breakdown for continuous microwave process at 100 tonne/year annual production

The results for the effects of extract yields obtained at different extraction temperature and time and solvent loading on the production costs are presented in Figure 7 below.

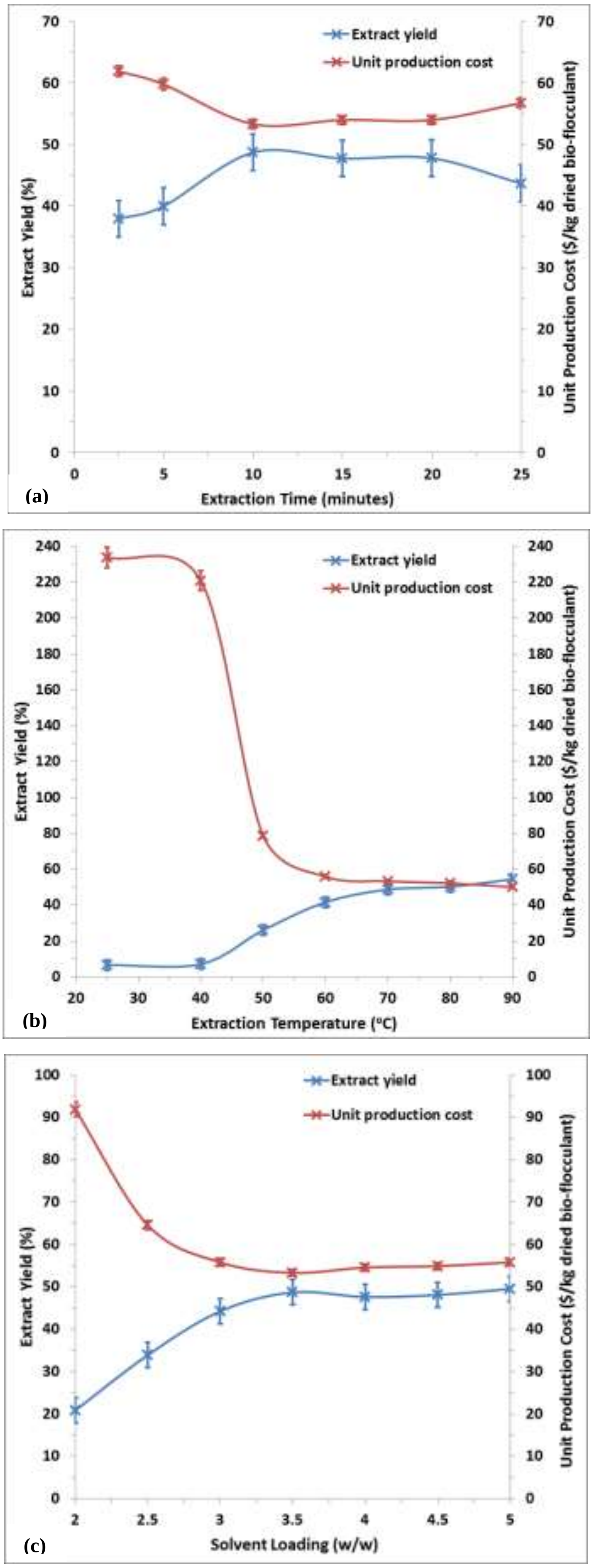
(a)
Extract yield
Unit production cost
Extract Yield (%)
Unit Production Cost ($/kg dried bio-flocculant)
0
10
20
30
40
50
60
70
0
5
10
15
20
25
Extraction Time (minutes)
(b)
Extract yield
Unit production cost
Extract Yield (%)
Unit Production Cost ($/kg dried bio-flocculant)
0
20
40
60
80
100
120
140
160
180
200
220
240
20
30
40
50
60
70
80
90
Extraction Temperature (°C)
(c)
Extract yield
Unit production cost
Extract Yield (%)
Unit Production Cost ($/kg dried bio-flocculant)
0
10
20
30
40
50
60
70
80
90
100
2
2.5
3
3.5
4
4.5
5
Solvent Loading (w/w)

Figure 7: The effects of extract yields with variations in (a) extraction time, (b) extraction temperature and (c) solvent loading to unit production cost

As shown in Figure 7(a), the extract yield was increased while the production cost was decreased with increasing of extraction time from 2.5 to 10 minutes. No significant change of production cost was observed between 10 to 20 minutes due to very minor change of extract yield within this extraction period. After 20 minutes of extraction time, the decrease of yield caused the rising of production cost. These results denoted the inverse relationship between extract yield and production cost. Similar finding was observed in Figure 7(b & c).

In the light of the same annual production, increase of extract yield directly reduces the requirement of raw material and utilities and manpower and also decreases the equipment size. As the result, the capital and operating costs are reduced and thereby decrease the production cost. Based on these results, it can be concluded that the optimised extraction conditions at 90˚C, 10 minutes and 3.5 w/w water loading provided the highest yield (54% on dry basis) with the lowest unit production cost ($50/kg dried bio-flocculant).

*3.2.3.2 Effect of raw material price*

The current okra price used in simulation was $1/kg. Another two possible scenarios were taken into consideration: (i) waste okra from market was used as the raw material with cost close to zero (Pereira, 2013), (ii) the market price of fresh okra was doubled ($2/kg) and the results are shown in Figure 8.

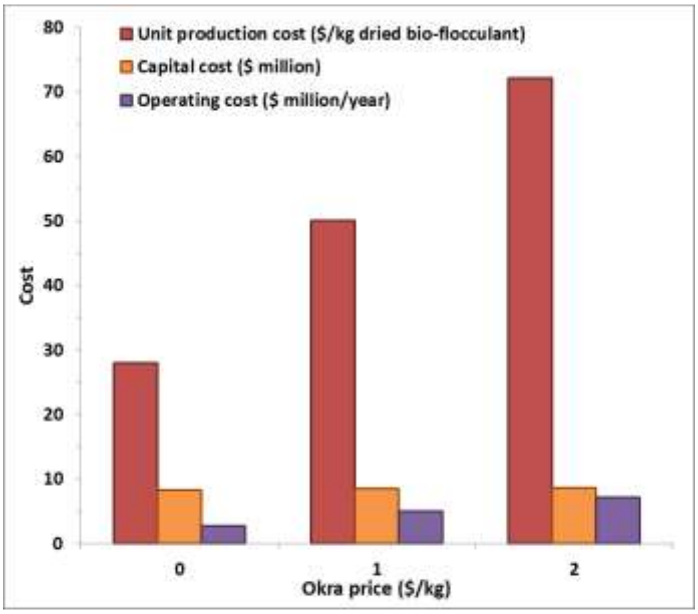

Figure 8: The effect of okra price on the capital, operating and unit production costs

It can be seen that okra price revealed high impact on the expenses of producing bio-flocculant, the operating and unit production cost were increased linearly with rising of okra price whilst the capital cost remained constant. From the standpoint of okra price at $1/kg, the operating and production costs were decreased by 44% if the okra was obtained for free and raised by 44% if the okra price was increased to $2/kg. Even though the extraction yield and the bio-flocculant quality of material produced from waste okra from the market is not verified yet, but it potentially presents a sustainable option to reduce the raw material cost and to resolve the problem of waste okra disposal.

### *3.2.4 Sensitivity analysis*

The results presented above revealed that the annual production and extract yield showed inverse relationship with the production cost. Meanwhile, the okra price demonstrated proportional relationship with the production cost. As fluctuations in annual production and extract yield and okra price showed impacts on the production cost, thus the sensitivities of these parameters to production costs were examined, and the results are presented in Figure 9.

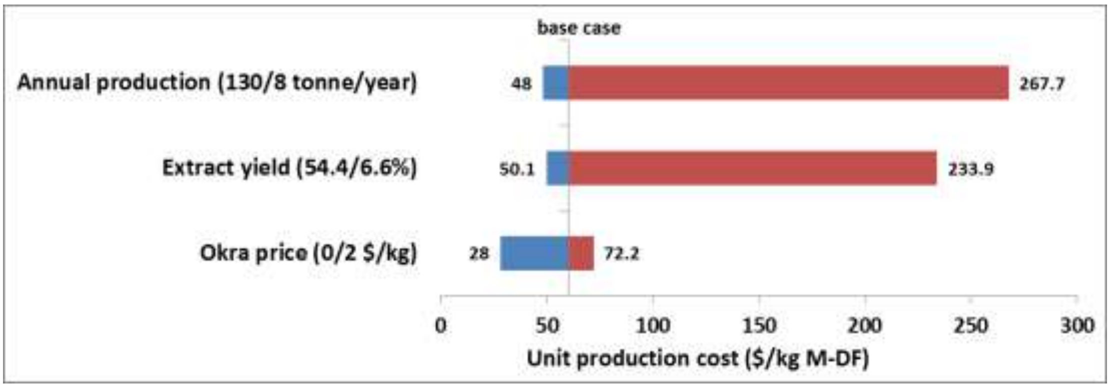

Figure 9: Sensitivity chart of unit production cost (The numbers in brackets on y-axis are the low and high values for each parameter.)

The range of each parameter from a low point (e.g. 6.6% for yield) to a high point (e.g. 54.4% for yield) was selected by referring to the results presented previously (Figures 5, 7 and 8). The unit production costs that correspond to the low and high points are shown. The chart shows that the production cost was most sensitive to annual production, followed by extract yield and least sensible to okra price. It is because the annual production and extract yield directly affect the production revenue. By increasing the production capacity, more bio-flocculants are produced and the revenues gained from selling of products could compensate the total capital and operating costs. Enhancement of yield reduces the requirement of okra and leads to reduction of raw material cost followed by lowering of operating cost. Hence, increasing the production capacity and investigation of modified extraction method/conditions that can increase the extract yield are the primary prerequisite to lower the production cost.

### *3.2.5 Profitability assessment of optimised production scheme*

Based on optimisation studies conducted above, continuous mode microwave process with extraction conditions at 90˚C, 10 minutes and 3.5 w/w solvent loading was identified as the optimised scheme for bio-flocculant production at the lowest unit production cost. The design specification and the purchase cost of each equipment for this proposed process at the annual production of 100 tonne/year is summarised in Table S2 within the supplementary material. Since okra bio-flocculant is a new product in the market and there is no similar product as a reference, the selling price was regulated to be 30 to 40% more than the production cost and the annual production was scaled up to 220 tonne/year in order to achieve profitable requirement. The profitability assessment results for optimised scheme at 220 tonne/year are listed in Table 7.

Table 7: Production and economic measures for the optimised scheme of bio-flocculant production

| **Process parameters** | | **Dried bio-flocculant** | **Aqueous bio-flocculant** |
|---|---|---|---|
| **Production measures** | Unit production cost ($/kg) | 37.3 | 31.4 |
| | Estimated selling price ($/kg) | 52.3 | 40.9 |
| **Economic measures** | Gross margin (%) | 28.6 | 23.1 |
| | ROI (%) | 22.2 | 25.1 |
| | Payback time (years) | 4.5 | 4.0 |
| | IRR (after tax, %) | 18.2 | 20.4 |
| | NPV at 7% ($) | $8.8 \times 10^6$ | $5.7 \times 10^6$ |

By referring to Table 7, the gross margins are positive and desirable which are 29 and 23% for dried and aqueous bio-flocculants respectively. It means that the company would retain $0.29/0.23 from each dollar of revenue generated as gross profit (Vučurović, 2012). In addition, the ROI values are in the expected range of 20 to 30%. Furthermore, the payback time is less than 5 years, which is acceptable. Most importantly, positive IRR and NPV are obtained which again show that this proposed scheme is realisable and economically viable.

### *3.2.6 Comparison of estimated bio-flocculant selling price with commercial flocculants*

The selling price of okra bio-flocculant is compared with the price of different commercial flocculants in Table 8.

Table 8: Price list for different types of flocculants

| Types of flocculants | | Price ($/kg) |
|---|---|---|
| Chemical flocculants | Cationic polyacrylamide (such as FO 4400 SH) | 4.25 (Long, 23 June 2016) |
| | Anionic polyacrylamide (such as AN 934 SH) | 4.08 (Long, 23 June 2016) |
| | Non-ionic polyacrylamide (such as FA 920 SH) | 4.08 (Long, 23 June 2016) |
| | Amphoteric polyacrylamide (such as FAM 4802) | 5.54 (Long, 23 June 2016) |
| Food grade bio-flocculants | Coconut shell activated carbon | 52 (Zhao, 4 July 2016) |
| | Chitosan | 50 (4 July 2016) |
| | Sodium alginate | 62.5 (2015) |
| Grafted starch-based flocculant | Amphoteric amylopectin | 191 (2016a) |
| Plant-based bio-flocculant from okra | Aqueous bio-flocculant | 40.9 |
| | Dried bio-flocculant | 52.3 |

The price of okra bio-flocculant is about 10 times higher than polyacrylamides. Yet, the downside of extra production cost could be balanced out by its considerable benefits to the environment and human health in circumstances where the use of polymeric flocculants is undesirable or prohibited. Most of the polyacrylamides are poorly biodegradable. Concerns have been raised that polyacrylamides employed in agriculture, food or beverage and water treatment sectors may contaminate the treated water or food products with acrylamide, a known neurotoxin (Shipp, 2006; Woodrow, 2008).

On the other hand, green bio-flocculant, which is extracted from renewable resources with water as solvent, is non-toxic and biodegradable. Hence, it poses minimum risk to human health and environment when compared to polyacrylamides. Due to these characteristics, bio-flocculants present a sustainable alternative to polyacrylamides for applications in food and beverage, cosmetic and pharmaceutical industries. The possible applications of bio-flocculants in different industry sectors have been discussed in previous publication (Lee, 2014). In addition, its price is shown to be lower than grafted starch-based flocculant and food grade bio-flocculants, which are widely used in beverage processing (Lee, 2014).

## 4. Conclusions

For production of industrial scale bio-flocculant, continuous mode MAE process with extraction conditions at 90˚C, 10 minutes and 3.5 w/w solvent loading was the optimised scheme that showed the lowest unit production cost. The production cost was strongly impacted by annual production and extraction yield, and moderately affected by raw material okra price. The selling prices of bio-flocculants with capacity of 220 tonne/year were lower than food grade bio-flocculants but higher than polyacrylamides. However, the sludge dewatering performance of microwave extracted bio-flocculant was verified to be comparable to or even better than polyacrylamides because higher water recoveries were achieved by bio-flocculant at the same dosage as polyacrylamides. For the sake of ecology and human health, okra bio-flocculant still represents a sustainable and economically viable alternative to chemical flocculants due to its high efficiency in sludge dewatering and environmentally friendly characteristic in terms of production and application.

## 5. Future Recommendations

The scaled up design process presented in this paper assumed that the bio-flocculant yield and quality will be the same as laboratory scale if the same extraction and operating conditions were used. However, it is obviously that the heat and mass transfer regime in the laboratory extractor is different to the industrial extractor. Hence, the next step is the design of larger scale continuous microwave extraction system for upgrading of bio-flocculant extraction to pilot scale or industrial scale. It has been shown that significant enhancements in yield can be achieved provided that microwave selective heating occurs (Lee, 2016), and this means that design of industrial microwave extractor will require further investigation/optimisation of the delivery of microwave power into the system to ensure the highest level of uniformity and efficiency of selective heating.

This study has shown that the production cost was decreased significantly by using waste okra as feedstock. Nonetheless, the investigation of flocculating and dewatering abilities of waste okra is very limited. A literature has reported the adsorption ability of okra waste for lead removal from aqueous solutions and proposed the application of okra waste for heavy metal removal from wastewater (Hashem, 2007). Therefore, the feasibility of using okra waste for bio-flocculant extraction should be justified in future work. As the capacity of okra waste in UK is expected to be small compared to the okra producing countries, it is suggested to move the production plant to India or Malaysia.

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## References

4 July 2016. Quotation for chitosan. G.T.C. Bio Corporation, China.
16 March 2016. Landfill tax: Increase in rates. HM Revenue & Customs, U.K.
21 June 2016. Frozen IQF okra. Crystal Rainbow Trading, South Africa.
22 January 2013. Generating energy from waste, including anaerobic digestion. Department for Environment, Food & Rural Affairs and Department of Energy & Climate Change, U.K.
2015. Quotation for sodium alginate. Modernist Pantry, U.S.
2015/2016. Scheme of charges. Severn Trent Water, U.K.
2016a. Quotation for amylopectin from maize. Sigma-Aldrich, U.K.
2016b. Statistical data set: International industrial energy prices. Department of Energy & Climate Change, U.K.
Anzanello, M.J., Fogliatto, F.S., 2011. Learning curve models and applications: Literature review and research directions. International Journal of Industrial Ergonomics 41, 573-583.
Athimulam, A., Kumaresan, S., Foo, D.C.Y., Sarmidi, M.R., Aziz, R.A., 2006. Modelling and optimization of Eurycoma longifolia water extract production. Food and Bioproducts Processing 84, 139-149.
Besaratinia, A., Pfeifer, G.P., 2007. A review of mechanisms of acrylamide carcinogenicity. Carcinogenesis 28, 519-528.
Bolto, B., Gregory, J., 2007. Organic polyelectrolytes in water treatment. Water Research 41, 2301-2324.
Brostow, W., Lobland, H.E.H., Sagar, P., Singh, R.P., 2009. Polymeric flocculants for wastewater and industrial effluent treatment Journal of Materials Education 31, 157-166.
Chung, H.S., Wibowo, S.Y., Fajar, B.K., Shin, Y.H., Jeong, H.M., 2012. Study on low pressure evaporation of fresh water generation system model. Journal of Mechanical Science and Technology 26, 421-426.
Coulson, J.M., Richardson, J.F., Backhurst, J.R., Harker, J.H., 1999. Chemical engineering: Fluid flow, heat transfer and mass transfer, 6th ed. Butterworth Heinemann, Oxford, p. 908.
Couper, J.R., Penney, W.R., Fair, J.R., Walas, S.M., 2012. Chemical process equipment: Selection and design, 3rd ed. Butterworth-Heinemann, Oxford, p. 801.
Fellows, P.J., 2009. Food processing technology: Principles and practice, 3rd ed. Woodhead Publishing Limited, U.K., p. 893.
Green, D.W., Perry, R.H., 2007. Perry's chemical engineers' handbook, 8th ed. McGraw-Hill Professional, p. 2703.
Harbourne, N., Marete, E., Jacquier, J.C., O'Riordan, D., 2009. Effect of drying methods on the phenolic constituents of meadowsweet (Filipendula ulmaria) and willow (Salix alba). LWT - Food Science and Technology 42, 1468-1473.
Harrison, R.G., 2015. Bioseparations science and engineering, in: Harrison, R.G., Todd, P.W., Rudge, S.R., Petrides, D.P. (Ed.), 2nd ed. Oxford University Press, U.S., p. 577.
Hashem, M.A., 2007. Adsorption of lead ions from aqueous solution by okra wastes. International Journal of Physical Sciences 2, 178-184.
Heinzle, E., 2006. Chapter 4: Sustainability assessment, in: Heinzle, E., Biwer, A.P., Cooney, C.L. (Ed.), Development of sustainable bioprocesses: Modeling and assessment. John Wiley & Sons, Ltd, U.K., pp. 81-117.
Heiwall, A., Calderon, V.J., Ogbonnaya, D., 2007. Pectic polysaccharides isolated from fruit pods of okra. Google Patents.
Hesami, F., Bina, B., Ebrahimi, A., 2014. The effectiveness of chitosan as coagulant aid in turbidity removal from water. International Journal of Environmental Health Engineering 2, 46-51.
Jenkins, S., 2016. Current economic trends - March 2016, Chemical Engineering: Essentials for the CPI Professionals

Katalin, B.B., Petra, C., Sándor, B., Zsófia, C., Cecília, H., 2012. Berry pectins: Microwave-assisted extraction and rheological properties. Food Bioprocess Technol 5, 1100-1105.

Kumar, S., Dagnoko, S., Haougui, A., Ratnadass, A., Pasternak, D., Kouame, C., 2010. Okra (Abelmoschus spp.) in West and Central Africa: Potential and progress on its improvement. African Journal of Agricultural Research 5, 3590-3598.

Lam, K.F., Leung, C.C.J., Lei, H.M., Lin, C.S.K., 2014. Economic feasibility of a pilot-scale fermentative succinic acid production from bakery wastes. Food and Bioproducts Processing 92, 282-290.

Lee, C.S., Binner, E., Winkworth-Smith, C., John, R., Gomes, R., Robinson, J., 2016. Enhancing natural product extraction and mass transfer using selective microwave heating. Chemical Engineering Science 149, 97-103.

Lee, C.S., Chong, M.F., Robinson, J., Binner, E., 2014. A review on development and application of plant-based bioflocculants and grafted bioflocculants. Industrial & Engineering Chemistry Research 53, 18357-18369.

Lee, C.S., Chong, M.F., Robinson, J., Binner, E., 2015. Optimisation of extraction and sludge dewatering efficiencies of bio-flocculants extracted from Abelmoschus esculentus (okra). Journal of Environmental Management 157, 320-325.

Leland Blank , P.E., Anthony Tarquin , P.E., 2011. Engineering economy, 7th ed. McGraw-Hill, New York, p. 642.

León-Martínez, F.M., Rodríguez-Ramírez, J., Medina-Torres, L.L., Méndez Lagunas, L.L., Bernad-Bernad, M.J., 2011. Effects of drying conditions on the rheological properties of reconstituted mucilage solutions (Opuntia ficus-indica). Carbohydrate Polymers 84, 439-445.

Levente, M., Anita, V., 2017. An estimation of the learning curve effect on project scheduling with calendar days calculation. Procedia Engineering 196, 730-737.

Liu, W.-C., Gong, T., Wang, Q.-H., Liang, X., Chen, J.-J., Zhu, P., 2016. Scaling-up Fermentation of Pichia pastoris to demonstration-scale using new methanol-feeding strategy and increased air pressure instead of pure oxygen supplement. Scientific Reports 6, 18439.

Long, R., 23 June 2016. Quotation for polyacrylamides. SNF (UK) Limited, U.K.

Neil, J.M., 2005. Project and cost engineers' handbook, in: Humphreys, K.K. (Ed.), 4th ed. Marcel Dekker, New York, p. 338.

Pereira, C.G., Prado, J.M., Meireles, M.A.A., 2013. Chapter 12: Economic evaluation of natural product extraction processes, in: Rostagno, M.A., Prado, J.M. (Ed.), Natural product extraction: Principles and applications. Royal Society of Chemistry, U.K., pp. 442-471.

Pirbalouti, A.G., Mahdad, E., Craker, L., 2013. Effects of drying methods on qualitative and quantitative properties of essential oil of two basil landraces. Food Chemistry 141, 2440-2449.

Richardson, J.F., Harker, J.H., Backhurst, J.R., 2002. Chemical engineering: Particle technology and separation processes, 5th ed. Butterworth-Heinemann, Oxford, p. 1208.

Robinson, J.P., Kingman, S.W., Snape, C.E., Bradshaw, S.M., Bradley, M.S.A., Shang, H., Barranco, R., 2010. Scale-up and design of a continuous microwave treatment system for the processing of oil-contaminated drill cuttings. Chemical Engineering Research and Design 88, 146-154.

Rodriguez-Jasso, R.M., Mussatto, S.I., Pastrana, L., Aguilar, C.N., Teixeira, J.A., 2011. Microwave-assisted extraction of sulfated polysaccharides (fucoidan) from brown seaweed. Carbohydrate Polymers 86, 1137-1144.

Saravacos, G.D., Maroulis, Z.B., 2011. Food process engineering operations, in: Sun, D.W. (Ed.). Taylor & Francis Group, LLC, Boca Raton, p. 586.

Seader, J.D., 2011. Separation process principles: Chemical and biochemical operations, in: Seader, J.D., Henley, E.J., Roper, D.K. (Ed.), 3rd ed. John Wiley & Sons, Inc., U.S., p. 849.

Sharma, B.R., Dhuldhoya, N.C., Merchant, U.C., 2006. Flocculants-An ecofriendly approach. Journal of Polymers and the Environment 14, 195-202.

Shipp, A., Lawrence, G., Gentry, R., McDonald, T., Bartow, H., Bounds, J., Macdonald, N., Clewell, H., Allen, B., Landingham, C.V., 2006. Acrylamide: Review of toxicity data and dose-response analyses for cancer and noncancer effects. Critical Review in Toxicology 36, 481-608.

Silla, H., 2003. Chemical process engineering: Design and economics. Taylor & Francis Group LLC, U.S., p. 483.

Sinnott, R.K., 2005. Chemical engineering design, 4th ed. Elsevier Butterworth-Heinemann, Oxford, p. 1055.

Thomas, N., 2016. Average hours worked by UK full-timers fell in 2015, Financial Times.

Towler, G., 2013. Chemical engineering design: Principles, practise and economics of plant and process design, in: Towler, G., Sinnott, R. (Ed.), 2nd ed. Butterworth-Heinemann, U.K., p. 1269.

Vučurović, D.G., Dodić, S.N., Popov, S.D., Dodić, J.M., Grahovac, J.A., 2012. Process model and economic analysis of ethanol production from sugar beet raw juice as part of the cleaner production concept. Bioresource Technology 104, 367-372.

Wei, X.L., Chen, M.A., Xiao, J.B., Liu, Y., Yu, L., Zhang, H., Wang, Y.F., 2010. Composition and bioactivity of tea flower polysaccharides obtained by different methods. Carbohydrate Polymers 79, 418-422.

Woodrow, J.E., Seiber, J.N., Miller, G.C., 2008. Acrylamide release resulting from sunlight irradiation of aqueous polyacrylamide/iron mixtures. Journal of Agricultural and Food Chemistry 56, 2773-2779.

Yeoh, S., Shi, J., Langrish, T.A.G., 2008. Comparisons between different techniques for water-based extraction of pectin from orange peels. Desalination 218, 229-237.

Zhao, T., 4 July 2016. Quotation for activated carbon. Zhengzhou Wade Water Treatment Material Co., Ltd., China.