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DESIGN AND SCALE-UP OF CONTINUOUS FLOW MICRO-REACTOR

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Abstract

Micro-reactor systems are a promising progress in chemical engineering. The challenge is to design continuous processes enable to develop products with uniform quality and high quantity,it might be provided by using the micro-reactors, the industrially important production of fine chemicals is frequently characterized by low conversions and yields, while the reaction conversion using the microreactors approaches +99%, which reduces the load in the separation step. The small volume of the micro-reactor issaving the material of construction, it isalso, enhanced the mass and heat management. Micro-reactors were locally designed and fabricated to develop a comprehensive study of micro-reactors performance. The aim of this article is the increasing of micro-reactor productivity through a numbering-up of the number of channels and micro-reactors. Multi-channel micro-reactors consist of ten and twenty Y-shaped channels areemployed. Esterification reaction of ethanol/2-ethyl hexanoic acid was taken as an example to test the developed micro-reactors, itcarried out using different designs of micro-reactors.The reactions were completed at temperature 25°C and molar ratio 1:1. The product flow rate is significantly increased (0.00028-2.5E-6 $m3s^{-1}$). A battery of 3-parallel micro-reactors of twenty channels was used to produce Ethyl Hexanoate, an economic feasibility study is developed.

Keywords: Feasibility, Fine chemical, Micro-reactor, Multi channels, Scaling-up.

1. Introduction

The state-of-the-art of micro-reactor technology has an excessive potential as a promising tool for reactor development and reactor scaling-down, it is one of the auspicious progressive know-how in a chemical engineering field, particularly in the production of a fine chemical. It may replace or partially replace the continuous large-scale reactors; hypothetically, it invents a breakthrough in reactor design and consequently in chemical engineering sciences [1, 2]. The miniaturization is used to define the scaling down in the equipment design, particularly in micro-channelling technology and microchemical engineering as the characteristic dimensions of the micro-equipment; the micro-channels stereotypically range from sub-micrometer to sub-millimeter range [3, 4].

The use of micro-reactor makes it probable for chemists and process engineers to achieve reactions with an unprecedented control over mixing, mass and heat transfer, safety, reaction residence time and other process parameters, which results in enhance reproducibility. In addition, they allow the specialists to utilize extreme reaction conditions (e.g., high temperature and pressure), in safe and reliable approach. In micro-reactors, the reaction rates can be accelerated and the reaction times can be reduced from hours to minutes and seconds, this development is recognized by miniaturization, which it became a new elaboration attitude in equipment design [5].

In the flow chemistry (sometimes referred to as a plug flow, microchemistry, or continuous flow chemistry), the reactants are continuously pumped through the reactor and the product is continuously collected, as realised in Fig. 1.

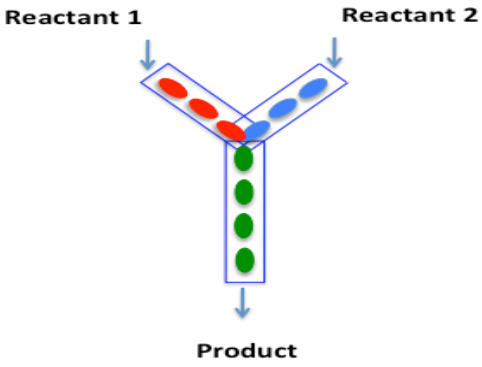


Fig. 1. The flow chemistry.

The significant factors in the classical reactor are reactants concentrations, mixing, temperature and reaction time (residence time), while in the micro-reactors, the factors affecting the process are residence time, mixing, temperature, and pressure. It is evident that the order of magnitude of the microchannel is in micron, therefore, the small fluid flow patterns lead to mixing times shorter than 100 μs (microsecond) in gases and approximately 1 μs in liquids, which is referred to the negligible resistance. Mixing time is only one criterion for an optimal chemical reaction; it is the scale of fluids residence time within the device [4, 5]. This is the main reason for the improved selectivity and high yield of chemical reactions in micro-reactors [5, 6].

The key advantages of Micro-Reactor Technology achieved in the research and industrial applications are exemplified in the surface to volume ratio, perfect mixing, and mass and heat transfer properties. It is also permitting to do mixing, heat transfer operations and chemical reactions in harmless environmentally friendly conditions. The micro-reactors have been offering high product quality; it leads to reduce the materials consumption and material of construction as well and also leads to high reaction conversion or selectivity advantages in different reactions types [7-16]. The principles of a conventional continuous stirred tank reactor and a continuous flow micro-reactor are compared in Fig. 2 [4]. Table 1 illustrates the difference between them.

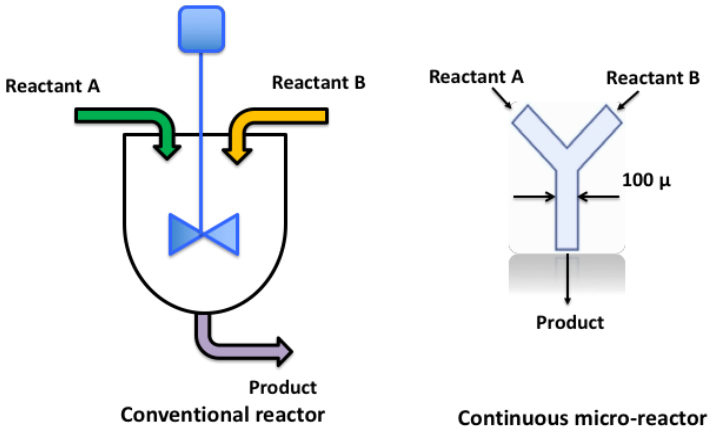


Fig. 2. Traditional CSTR and continuous flow micro-reactor [4].

Table 1. Conventional CSTR and continuous micro reactor.

Conventional CSTR	Continuous micro-reactor
Classic Reactor	New trend
Reactants are fed continuously, mixed, and the product is getting out continuous	The reactants are fed continuously
Continuous operation	Reactants are mixed in flow channel.
Good temperature control	Perfectly mixed
Good control	The products are continuously collected
Simplicity of construction	High conversion per unit volume
Lowest conversion per unit volume	Scale-independent synthesis
By-passing and channelling possible with poor agitation	Product profile improvement
	Accelerated process development
	Enhanced safety
	Constant product output quality
	Cleaner product profile
	Higher yields

Some researchers indicated the using micro-reactor technology in fine chemicals and pure biodiesel production; it might be produced by a micro-reactor of a size comparable with conventional scales, besides that, the reaction might occur at mild conditions, the residence time was about 4 min; over 99 % yield. As an example, recently, the yield of methyl esters of 99.4 % is obtained at the residence time of 5.89 min with KOH concentration of 1 % and at 60 °C in microcapillary micro-reactor with the inner channel diameter of 0.25 mm [16]. Thus, it could be concluded that higher methyl ester yield is obtained at even shorter residence time.

The challenging problem in micro-reactors is how to increase the throughput, which is considered a major problem in the micro-reactor applications; therefore, the numbering-up either of the channels or the reactors might be an excellent solution [17]. A simplified scheme illustrating scale-up versus numbering-up (channelling and micro-reactor) strategies can be appreciated in Fig. 3, where *A* and *B* are the reactants while *C* is the product. [17-19].

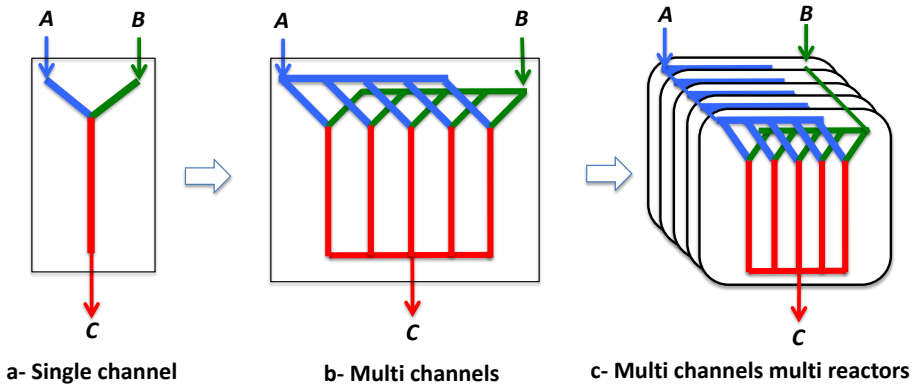


Fig. 3. Micro-reactor scaling-up.

The aim of this work is to increase the micro-reactor productivity; therefore, single, ten and twenty channels micro-reactors are developed and implemented, in addition, scaling-up using three micro-reactors of multi-channels (20 channels), which is elaborated to achieve high production capacities. Moreover, production of the fine chemical component will be taken as an example to study the performance of the micro-reactors technically and economically.

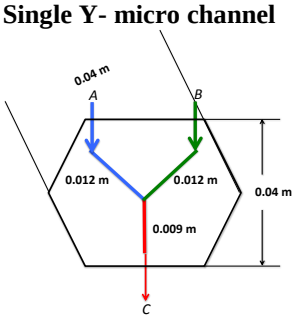
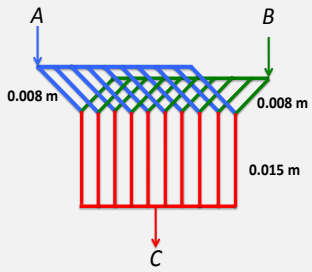
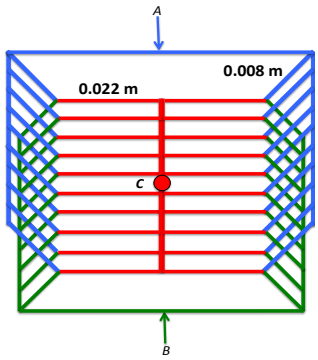
2. Experimental work

2.1. Design of multi channels micro-reactors system

Three micro-reactors are designed particularly to study the effect of channel numbering-up on scaling-up of the production rate. Therefore, single, ten and twenty channels micro-reactors are designed, manufactured and used to produce Ethyl-ethylhexanoates.

Table 2 illustrates the general characteristics and dimensions of three designed prototypes.

Table 2. General characteristics and dimensions of three designed prototypes.

Type of micro-reactor	Description	Dimensions
Single Y- micro channel 	A hexagonal block of two parts (bottom and top) of stainless steel 316 L, with three opening ports; two are reactants inlet nozzles and one is product outlet nozzle, it has a single Y-shape channel, the micro-channels are completed using the Laser technique.	Channel diameter; *d;* 100 μm, height, *h;* of the hexagonal block is 0.04 m, each part of the block of 0.02 m, each port of 0.005 m diameter, *d*. The length, *l*; of the 2 legs of Y-shape is 0.012 m, and the length of a vertical leg is 0.009 m.
Ten Y- micro channels 	A squared block of stainless steel 316 L, consists of 2 parts (bottom and top), has three opening ports; two reactant inlets, each port is connected to a duct supplying the reactants. One product outlet nozzle is connected to holding duct collecting the product from each channel; the micro-channels are completed using the Laser technique.	Channel diameter; d; 100 μm. A block of a squared shape of 0.07*0.07 m^2. Block depth 0.04 m. Each part of the block of 0.02 m. Each port nozzle 0.005 m in diameter. The length of the 2 legs of Y-shape is 0.008 m, and the length of the vertical leg is 0.015 m.
Twenty Y- micro channels 	A rectangular block of stainless steel 316 L, consists of 2 parts, has three ports; 2 reactants inlet nozzles, each port is attached to 2 ducts, each duct is connected to 20 channels, to supply the reactants. One product outlet is connected to holding point collecting the product from the 20 channels; the micro-channels are developed using the Laser technique.	Channel diameter; 100 μm. Rectangular shape of 0.057 m width, 0.082 m length and 0.04 m depth. Each part of the block of 0.02 m. Each port nozzle 0.005 m in diameter. The length of the 2 legs of Y-shape is 0.008 m, and the length of the vertical leg is 0.022 m.

2.2. Design of pilot system of micro-reactors

A pilot system comprised three micro-reactors, each has twenty channels, are implemented and operated in parallel.

2.2.1. The detailed design of the reactor

Description of the reactor

The reactor is a rectangular block of stainless steel 316 L, consists of 2 parts, has three ports; 2 reactants inlet nozzles, each port is attached to 2 ducts, each duct is

connected to 20 channels, to supply the reactants. One product outlet is connected to holding point collecting the product from the 20 channels.

The main dimensions of the reactors are the following: Channel diameter (100 μm), Length × width × depth (0.057 m × 0.082 m × 0.04 m), Port nozzle (0.005 m diameter), the length of the 2 legs of Y-shape (0.008 m), and the length of vertical leg (0.022 m).

2.2.2. The pilot unit

The pilot system is comprised of three parallel micro-reactors, attached to feeding peristaltic pumps for supplying the two reactants throughout two headers. The system has two feeding tanks and product tank, these tanks are of Stainless steel.

Figure 4 demonstrates the schematic process flow diagram of the pilot system.

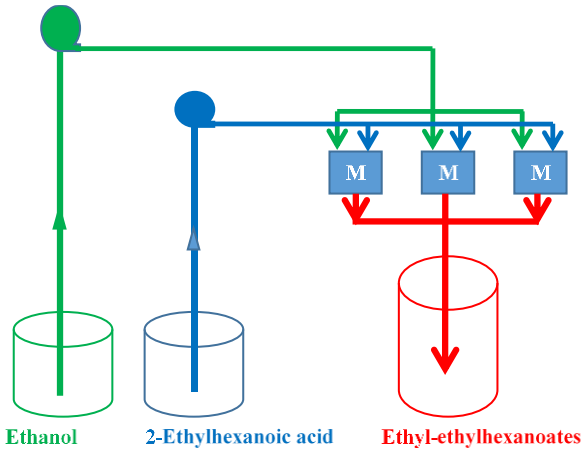


Fig. 4. Schematic diagram of pilot system.

2.3. Ethyl Ethylhexanoate production

Esterification of 2-Ethylhexanoic acid produces compounds of great important (ethylhexanoate) in pharmaceutical and food industry. Such product is an aniseed, red apple, fruity apple or ester flavour compound [20]. The esterification reaction is conducted using all designed systems. A series of experiments are performed using the developed micro-reactors.

The experiments are completed at fixed operating conditions; molar ratio 1:1 (2-Ethylhexanoic acid/Ethanol) at 25°C, feed flow rates are: 0.015E-6 and 0.0062E-6 m^3s^{-1} for 2-Ethylhexanoic acid and Ethanol respectively for single Y-channel micro-reactor (First prototype), at 0.56E-6 and 0.2E-6 m^3s^{-1} for 2-ethylhexanoic acid and Ethanol respectively for the ten and twenty Y-channels micro-reactor (Second and third prototypes).

Testing the three parallel micro-reactors of 20 channels (Pilot System) is developed. Three units of micro-reactor of twenty channels were operated in parallel to scale-up the production of micro-reactors.

One experiment using the developed pilot system was conducted to investigate the esterification reaction of 2-ethylhexanoic acid/ethanol at molar ratio 1:1 and

25°C, at feed flow rates of 1.85E-6 and 0.56E-6 m^3s^{-1} of 2-ethylhexanoic acid and ethanol respectively.

3. Results and Discussion

3.1. Reactor design and implementation

The process and mechanical designs of the three prototypes of micro-reactors are developed and implemented; the designs are bidding, procuring and manufacturing. These micro-reactors are fabricated in a local private manufacturing plant (Hasco). They are manufactured from Stainless Steel 316L using Laser technique; Figs. 5-7 illustrate the detailed mechanical drawings of single, ten and twenty channels micro-reactors respectively.

Figure 8 demonstrates the manufactured three prototypes of micro-reactors.

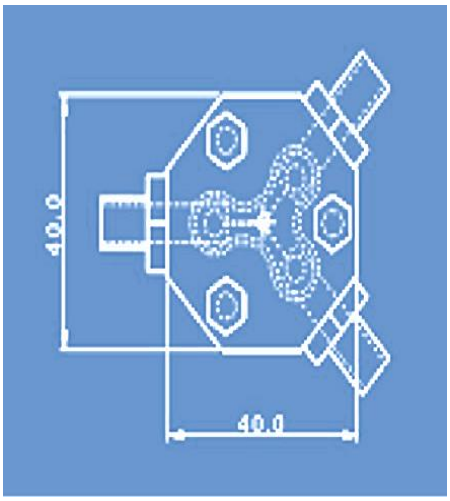


Fig. 5. Detailed mechanical drawing of single channel microreactors.

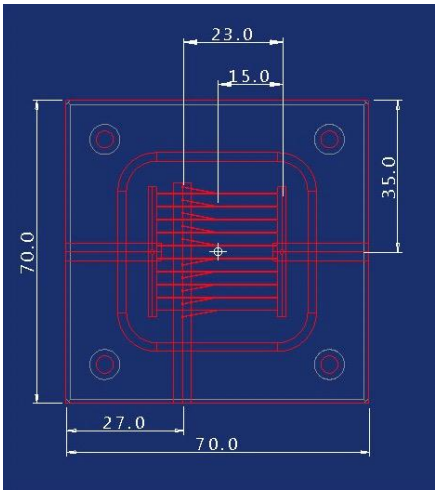


Fig. 6. Detaled mechanical drawing of ten channels microreactor.

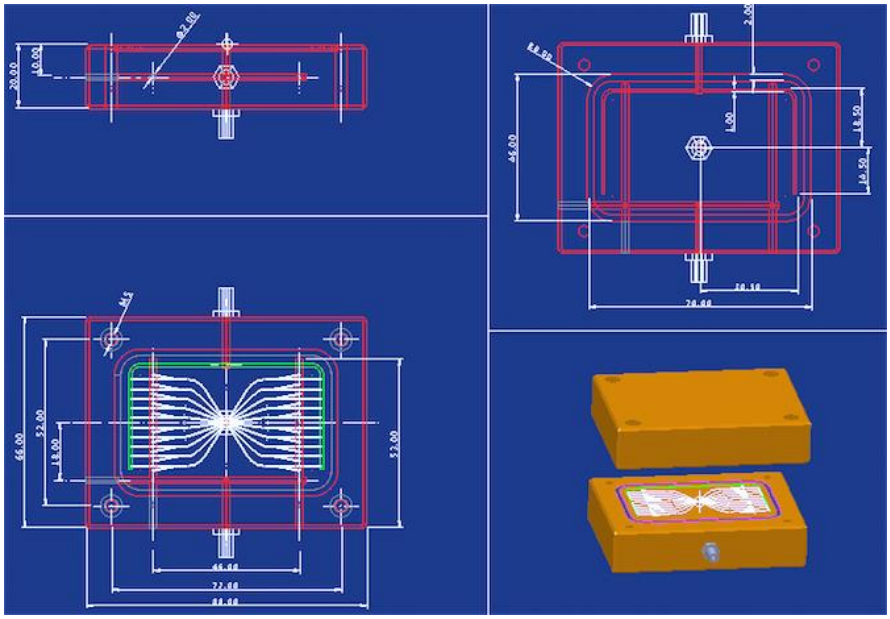
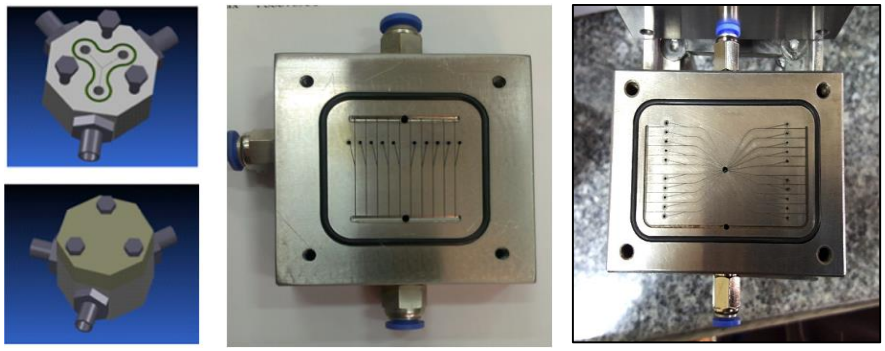


Fig. 7. Detaled mechanical design of tenty channels microreactor.



a- Single channel **b- Ten channels** **c- Twenty channels**

Fig. 8. Manufactured micro-reactors (three prototypes).

3.2. Testing and operation

Esterification reactions are studied to assess and evaluate the reactor performance. Esterification of 2-ethylhexanoic acid/ethanol to produce Ethyl-2-ethylhexanoate is accomplished at molar ratio of 1:1 and at 25°C.

The residence time $= \frac{\text{Volume of reactor}}{\text{Flow rate of reactants}}$ [6].

Table 3 depicts the pumping flow rates of the two reactants, reactor volume, integrated reactants flow rate, reaction residence time, reaction conversion and the product flow rate of single, ten and twenty channel reactors.

It is clear from Table 3 that the reaction conversion is nearly the same in all microreactors alternative, which may be attributed to the perfect mixing in the very

narrow channels, where the resistance is negligible. It is also noticeable from Table 3 that the reactor productivity is significantly increased, where the product flow rate is increased from 0.00028E-6 to 2.5E-6 m^3s^{-1}, which may be referred to the numbering-up of channels and increasing the flowing quantities.

Table 3. The operating conditions and results of the three micro-reactors.

Reactor type	Feed flow rate, *q*, m^3s^{-1}		Reactor Volume, m^3	Residence *t*, Time, μs	Reaction conversion	Product flow rate, m^3s^{-1}
	Acid	Ethanol				
Single Y channel	0.15E-7	0.6E-8	0.53E-10	2.456	0.993	0.28E-9
Ten Y channels	0.56E-6	0.2E-6	0.243E-8	3.200	0.998	0.875E-6
Twenty Y channels	0.56E-6	0.2E-6	0.597E-8	7.416	0.997	2.5E-6

3.3. Pilot micro-reactors system

A pilot system of three micro-reactors of twenty channels is developed and connected in parallel; two feeding peristaltic pumps are used to supply the two reactants. The three-micro-reactor are operated at molar ratio 1:1; 2-ethylhexanoic acid/Ethanol and at 25°C, the pilot system is demonstrated in Fig. 9.

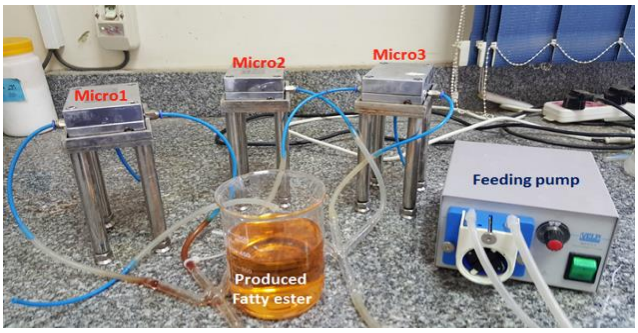


Fig. 9. Pilot system of three parallel 20 channels microreactor.

The reaction conversion using the designed pilot system is reached 0.994 and the production volume is 0.450E-3 m^3 after 180 s, i.e. 0.132E-3 m^3s^{-1}. The numbering-up of channels with numbering-up of micro-reactor leads to significant increase of the production rate and reduce the residence time of reaction.

4. Feasibility study and economic evaluation

4.1. Conceptual Design

The economic evaluation of the contemporary process is developed. It is based on the results of a pilot experiment using three parallel micro-reactors each has twenty channels. The process design is established on the following principles:

i. Develop a production facility of 56.42 t/year capacity. This assumption is based on the results of the pilot experiment.

ii. The designed facility will be working 330 d/year, 24 h/day for 3 shifts of 8 h.

iii. Bank of continuous three reactors with the same size will be used to operate in parallel; the block diagram of this process is depicted in Fig. 10.

iv. The three reactors are fed with reactants in parallel using two centrifugal pumps one for alcohol while the other for 2 methyl hexanoic acid.

v. The product mixture (water and 2-ethylhexanoate) will be addressed to a distillation column to separate the product. The water (BP; 100°C) and unreacted Ethanol (BP; 78.37°C) are collected at the top of the column, while the product Ethyl Hexanoate (BP; 168°C) is collected from the middle of the column, while the Ethyl hexanoic acid (BP; 228°C) is collected in the bottom of the column.

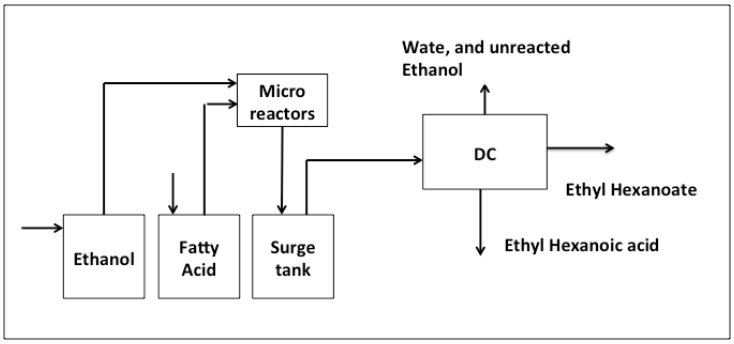
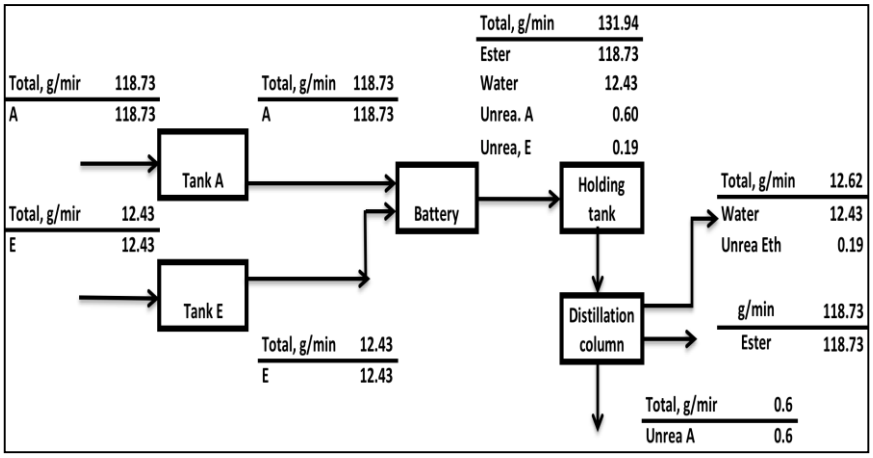


Fig. 10. Block diagram of pilot unit of micro-reactors.

4.2. Mass balance

The mass balance was developed based on the experimental results of the pilot experiment; it is illustrated in Fig. 11.



where: A= 2 Ethyl hexanoic acid, E= Ethanol, Ester= Ethyl Hexanoate

Fig. 11. Mass balance flow sheet, basis: 1 min.

Figure 12 illustrates the process flow diagram of the production of Ethyl Hexanoate using a battery of three micro-reactors.

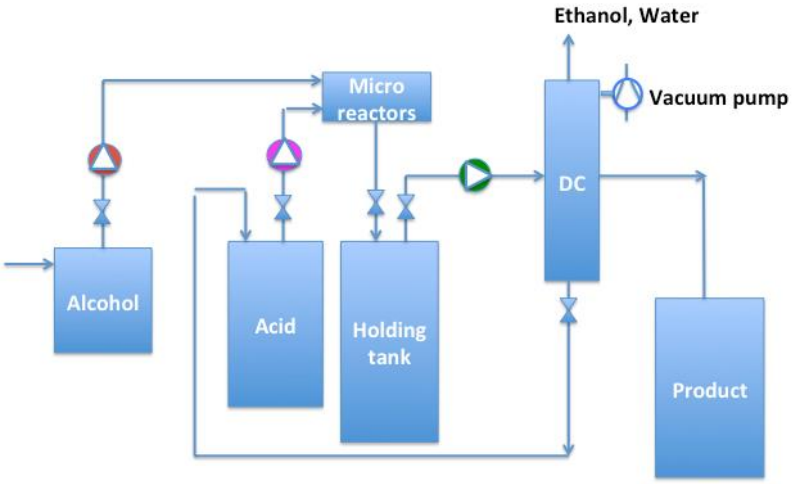


Fig. 12. Process flow diagram.

4.3. Equipment design and specifications

Below are the equipment design and specifications

- A cylindrical stainless steel 316 L vessel, of size 0.07 m^3 of 0.40 m diameter and 0.6 m height stores Ethanol for 8h.
- A cylindrical stainless steel 316 L vessel, of size 0.3 m^3 of 0.6 m diameter and 0.9 m height stores 2-ethylhexanoic acid for 8h.
- A cylindrical stainless steel 316 L vessel of 0.7 m diameter and 1.0 m height is used to hold reaction product mixture for 8h; its size is about of 0.4 m^3. A product tank same as the holding tank is used to collect the product, same as holding tank (3).
- Two feeding pumps for 2-ethylhexanoic acid and methanol to the reactors, and then another one to feed the product mixture to distillation column, and a vacuum pump which is attached to the distillation column to operate under vacuum.
- Three micro-reactors are working in parallel; each reactor has 20 micro channels.
- The distillation column is working at 50^{o}C under vacuum.

4.4. Capital Investments

4.4.1. Fixed cost

The equipment costs are determined according to published costs of stainless steel 316 L of 2016; $ 2094/t. Table 4 illustrates the equipment cost of the integrated system. While Table 5 depicts the fixed cost of the developed facility.

4.4.2. Annual cost

Table 6 depicts the annual costs; the electricity cost is estimated on the local governmental price LE 0.4, the maintenance is estimated by 7% of the equipment, the services is estimated as LE 100000, the depreciation is anticipated as 10% of the equipment cost plus 20% of the contingency, an amount is allocated for other emerging items as 10% of the total annual costs.

4.4.3. Production Cost

The annual cost of the unit product is equal to the annual costs/annual production; therefore; it is equal LE 24765/ton Ethyl 2-ethylhexanoate; i.e. $ 1.37/kg (where; 1$ equal LE18.05), while the international selling price is **$**1-5/kg**.** Therefore, the produced ethylhexanoate is promising to be a competitor with respectable benefit margin.

Table 4. Equipment and costs.

Code	Item	No. Piece	Price/unit, LE	Total price, LE
1	Storage tanks	4	_	13772
2	Pump of methanol	1	5000	10000
3	Pump of Fatty Acid	1	10000	15000
4	Pump of product	1	10000	15000
5	Vacuum pump	1	20000	25000
6	Micro reactor	3	10000	50000
7	Distillation column	1	100000	200000
Total				**328772**

Table 5. Fixed costs.

Code	Item	% E	Value, LE
1	Equipment	E	328772
2	Freight and Shipping	**20**	65754
3	Equipment erection	**25**	82193
4	Measurement/control instruments	**20**	65754
5	Piping	**30**	98632
6	Electrical instruments and cables	**30**	98632
10	Others	40	131509
11	Contingency	10% total fixed cost	96805
Total			**968051**

Table 6. Annual costs.

No.	Item	Annual cost; LE
1	Raw materials	968051
3	Electricity	5523
4	Maintenance	67764
5	Services	100000
6	Depreciation	116166
8	Others	139723
Total		**1397226**

5. Conclusions

A series of micro-reactors were designed and fabricated in private local factory to complete a comprehensive study to reveal the behaviour and performance of these reactors and their importance in the field of chemical engineering.

Three micro-reactors are designed, implemented and used to realize this study. The numbering-up of the micro-channels was accomplished to increase the rate of production. The multi-channels micro-reactor consists of ten and twenty Y-shape channels lead to increase the production rate of the esterification reaction ten and twenty times compared by single Y-channel micro-reactor. Esterification reactions of ethanol/2-ethyl hexanoic acid were tested using the micro-reactors; all reactions were completed at temperature 25°C molar ratio 1:1. The product flow rate is significantly increased (0.00028-2.5E-6 m^3s^{-1}).

The feasibility study reveals the production cost of Ethyl 2-ethylhexanoate using the developed micro-reactor system is $ 1.37/kg, while the international selling price is **$**1-5/kg. Therefore, the produced ethylhexanoate is promising to be a competitor with respectable benefit margin.

Nomenclatures

d	Channel diameter, μm
h	Height of the hexagonal block, m
l	The length of block, m
q	Flow rate, m^3s^{-1}
T	Temperature, ^{o} C
t	Time, s and μs

Abbreviations

A	Acid (reactant)
B	Alcohol (reactant)
BP	Boiling Point
C	Ester (product)
LE	Egyptian Pound (Egyptian currency)
MR1	Microreactor 1
MR2	Microreactor 2
MR3	Microreactor 3

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