

# Flow visualization and flow-pattern mapping in coiled flow inverters for a liquid-liquid reaction system.

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#### Highlights

- Oil conversion increase as Dean number increases for slug flow.
- Oil conversion decreases under parallel flow despite an increase in Dean number.
- Dimensionless Flow Map for the intensification of Biodiesel production is presented.
- Flow-Mapping gives insight of suitable conditions for continuous Biodiesel production.

#### **1. Introduction**

Coiled Flow Inverters (CFIs) have been successfully used to enhance mass and heat transfer with applications such as mixers, heat exchangers, separators and single phase chemical reactors [1-2]. For liquid-liquid reaction systems such as Biodiesel production in CFIs, results showed that the reaction can be carried out twelve times faster than the conventional batch reactor and has the capability of decreasing methanol excess used in the reaction by 50% while achieving oil conversions of 90% [3]. These results are direct consequence of three main phenomena: 1) the interaction between the liquid-liquid interfaces of the reactants, 2) the presence of secondary flow (Dean flow) in curved geometries and 3) the inversion of the internal velocity profile at the 90° bends. This work explores the relationship between these phenomena by making direct flow-visualization in a CFI (Biodiesel production as case of study) to obtain a dimensionless flow-map (DFM) that give us insight of the most suitable flow conditions for liquid-liquid systems in CFI.

#### 2. Methods

Flow visualization was carried out in a glass CFI which consists of helical coiled tubes (OD~6.3 mm; ID~3.1 mm; curvature ratio,  $\lambda$ ~5) mounted vertically with equally spaced 90° bends (3 bends for the system tested) along the length of the reactor. Sunflower oil and methanol were fed into the system at different flow rates and reactants volumetric ratio (1 mL/min<Q<sub>oil</sub><21 mL/min; 1 mL/min<Q<sub>net</sub><17 mL/min) by two positive displacement pumps (Watson Marlow 120s). Photograph were taken using camera with high definition sensor (Sony  $\alpha$  a5000 with a Sony lens SELP165016-50mm) connected to a computer for video and photograph analysis. Photographs were taken at the inlet of the CFI (T connector OD~6.3 mm), at the coiled arm (4 turns in each arm) and at the 90° bend (inversion). The schematic diagram of the experimental set-up is shown in Figure 1. Weber numbers (*We*) were estimated with the flow conditions at the entrance of the system (average velocity of each reactant fed), while Dean numbers (*De*) were estimated at the coiled-arm flow conditions (total average velocity of the system).

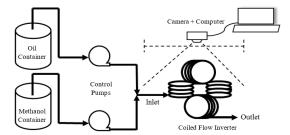


Figure 1. Experimental set-up for flow visualization in CFIs.



### 3. Results and discussion

Based on the large data base (3,125) different flow regimes were identified. Table 1 shows that with an increase in flow rate (at the same residence time) and number of inversions, oil conversion increases under Slug Flow pattern (SF), however, with a further increase in flow rate (Qtot~20 mL/min), the benefits of flow inversion and high radial mixing due higher values of De are overshadowed by a decrease in the overall specific surface area due to the presence of Parallel-Wavy Flow pattern (PWF). It is important to take into account the contribution of centrifugal forces to each phase, i.e., the viscosity of methanol is lower than oil, resulting in higher Reynolds numbers (*Re*) and thus higher *De*, increasing radial mixing within the components. This analysis will be more relevant in SF since the methanol phase is contained between the oil phase.

Table 1. Experimental results, dimensionless numbers and flow patterns for Biodiesel production in CFIs.

Q [mL/min]	Residence Time [min] [3]	Oil Conversion [%] [3]	Number of 90° Bends	We oil [-]	We methanol [-]	De oil [-]	De methanol [-]	Observed Flow Pattern
5	1	54	0	9.78E-02	2.60E-03	0.11	25.71	Slug Flow
7.5	1	68	1	1.86E-01	9.14E-03	0.20	37.36	Slug Flow
10	1	72	3	3.02E-01	1.62E-02	1.33	48.52	Slug Flow
20	1	64	7	1.36	6.50E-02	2.66	97.05	Parallel-Wavy Flow

Figures 2A-2C show the flow-maps for Biodiesel production in CFI. It can be seen that SF is the predominant flow pattern at the experimental conditions within a range of:  $0.001 < We_{oil} < 1$  and  $0.002 < We_{met} < 0.06$  increasing mass transfer between reactants due to the internal circulations inherit from this flow pattern and the effect of the inversions. However, with an increase in flow rate, an increase in the intensity of Dean flow increases and the flow pattern shifted from SF to PF and PWF (Q>18 mL/min,  $We_{oil} >= 1$  and  $We_{met} >= 0.06$ ), decreasing the overall specific surface area of the system and therefore the overall performance of the system. Figure 2C shows the ranges of We for each flow patterns observed in the system as well as the effect of flow inversion in PF.

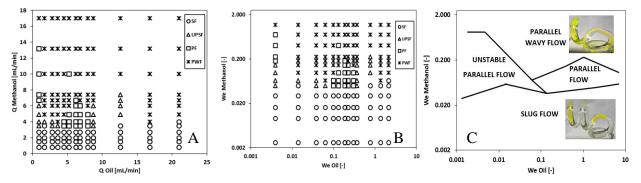


Figure 2. A) Flow-Map at different flow rates; B) Dimensionless Flow-Map for Biodiesel production: C) Operational ranges for different flow patterns in CFIs for Biodiesel production.

# 4. Conclusions

The overall performance of CFIs for Biodiesel production increases under SF with an increase in flow velocity and decreases under PWF. DFM for biodiesel production are useful tools for designing intensified equipment for continuous immiscible liquids processing. It is expected that the present study will further strengthen the design of CFI for liquid-liquid reactions.

# References

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#### Keywords

Slug-Flow; Biodiesel; Intensification; Liquid-Liquid Reaction