Solving Complex Problems in Formulation Processing by Building a Pragmatic Multi-Scale Model

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Abstract

A methodology for building pragmatic multi-scale models to solve complex problems is presented. The methodology is exemplified using a manufacturing process for a lyotropic liquid crystal product. The methodology generated a model that could be validated using specifically designed experiments. As a result it was discovered that spontaneous emulsification occurs during the process (which was previously unknown); this could be a crucial design factor in implementation of a manufacturing system. Further experiments had shown that by changing the contact concentration ratio between the materials, the viscosity of the product changes by a factor of two.

Keywords: Multi-scale, pragmatic model, structured fluids

1. Introduction

Traditional modelling techniques tend to model processes using complex mathematical formulae and equations (Noro *et al.*, 1999). However, it is often not practicable to employ such techniques when dealing with highly complex systems as it would be expensive, time consuming and most importantly for many such systems the underpinning physics and quantitative data required for the models to work are lacking (Prausnitz, 1998).

Traditional modelling tends to concentrate on either the molecular level (performed by chemists) or the macroscopic level (performed by engineers) of the problems and there is a general lack of linkage between them.

2. Methodology

The approach adopted in this project is to build a pragmatic model by using the observed phenomena and basic physics and science which are known to exist in the system. The completed model should be able to explain the phenomena observed on the system. The credibility of the model will be tested with specifically designed experiments. The model will also be used as a guide to design specific experiments to further explore the system and could potentially be used as a basic for any future mathematical model. This methodology will be exemplified using a manufacturing process typical of personal care product which involves a lyotropic liquid crystal (Holmberg, 2001).

Figure 1 shows the overall flowchart of the pragmatic modelling and experiments process.

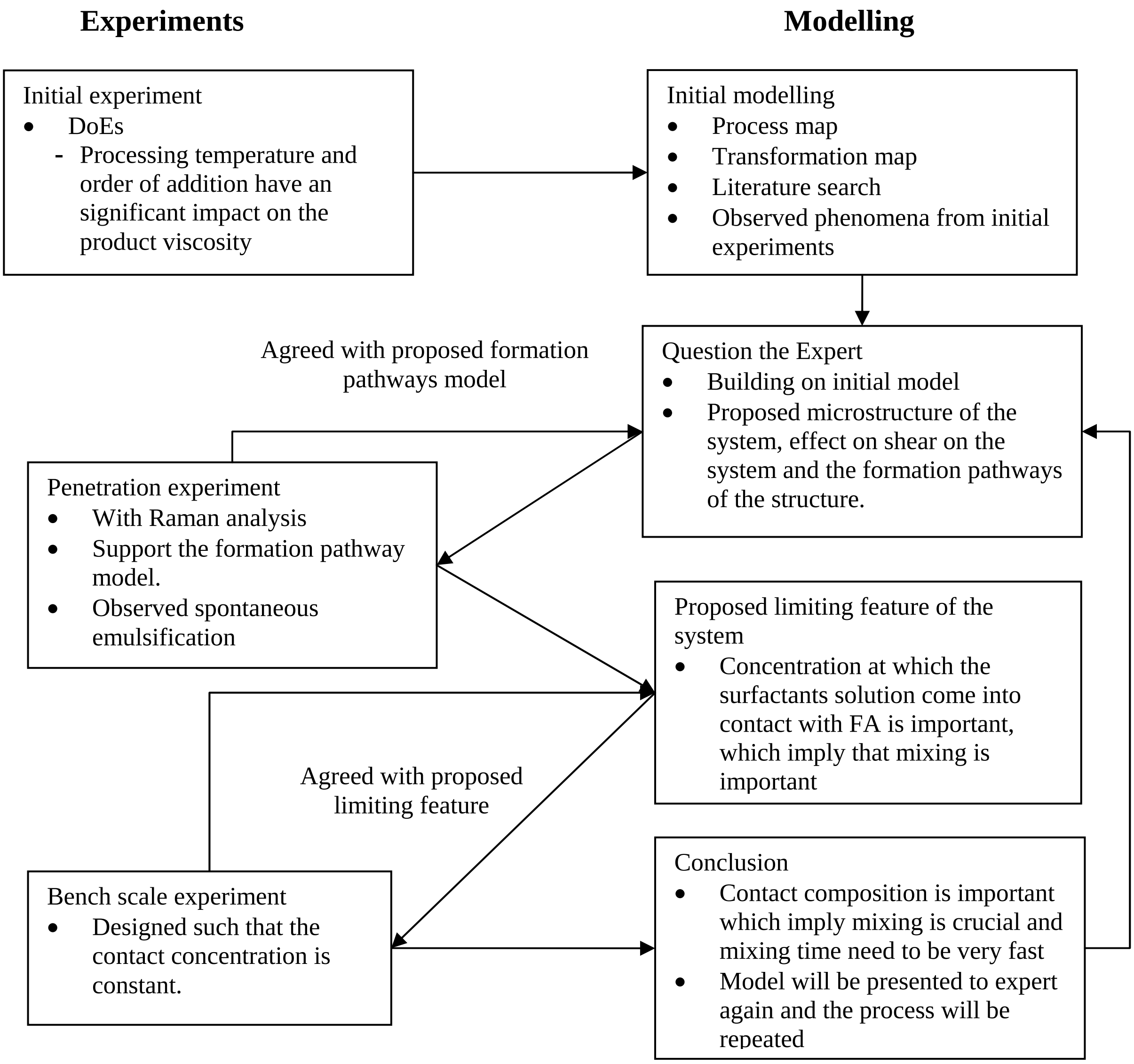

Figure 1 Overall flowchart of the modelling process

2.1. System and Manufacturing Process

The product consists of a mixture of cationic surfactants, long chain fatty alcohol and water. The manufacturing process is a typical hot batch process whereby the materials are added one after another with intense mixing and the addition of fatty alcohol occurring in an external recirculation loop. During the cooling process, the system experiences a phase transformation whereby it changes from a lamellar phase (L_α) to gel phase (L_β). At the end of the process, a viscosity measurement was taken as a form of quality control parameter.

2.2. Initial Experiments

The preliminary step of the methodology is to obtain any available information from the literature and to perform some experiments to have a overview on how the system behaves under different conditions.

A number of Design of Experiments (DoE) were designed and performed to investigate the various physical variables; processing temperature, agitators speed, cooling rate, order of addition of material and mixing time had on the system. It was concluded that processing temperature and order of addition had a significant impact on the product viscosity.

2.3. Initial Pragmatic Modelling

Using the knowledge gained from the initial experiments, known published literature and basic sciences of the system, an initial pragmatic model for the process was developed. The model initially consisted of a “transformation map” and a high level “process map”.

A process map is a consolidation of all the conditions, eg mass and heat transfer, shear field etc, the science involved, eg rate of formation, diffusion process, the physical variables which affect the conditions and the physical properties of the fluid. It also shows the links between the physical phenomena involved and the macro and micro properties of the system.

Figure 2 shows the process map of the manufacturing process. It captured the key variables and the sciences involved in the process and the linkage between them. This would provide a high level overview of the variables, conditions and science involved.

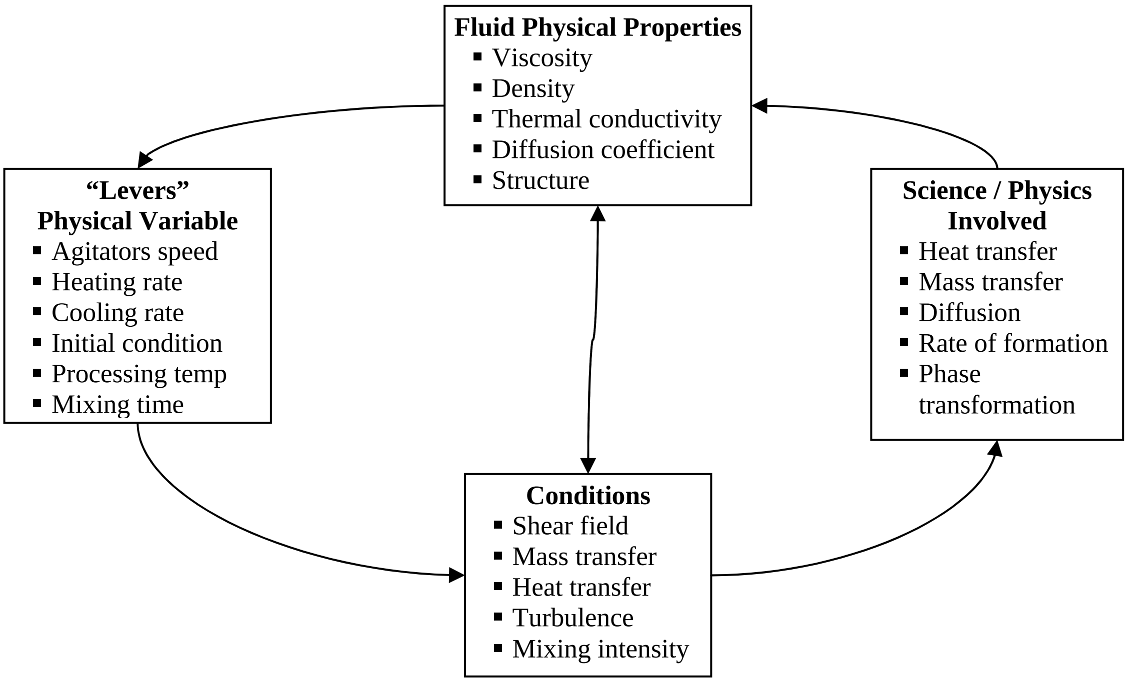

Figure 2 High level process map showing the relationship between the process related entities

A transformation map is a map of the transformations that the materials will experience during the manufacturing process. The transformation map is useful as it enables the process to be broken down into smaller parts and enables the identification of the most critical part of the process. It also enables us to attribute the relevant science to the relevant part, thus any gaps in the knowledge can also be identified

Figure 3 shows a transformation map of the system experiencing through a typical manufacturing process. From Figure 3 it can be seen that the key transformation step of the process is when the fatty alcohol and the surfactants solution are first mixed together. Despite being the key step part of the process, no information on the underlying science and processes that is taking place on this particular system are available in the public domain.

2.4. "Question the Expert"

The "Question the Expert" step is a structured discussion session with all the stakeholders of the process; this included the scientists who were working on a micro level and the engineers who were working on the macro level. There is a common trend in the processing industry whereby individual experts, who own the specific part of the process, have their own unique beliefs on how the system behaves and they tend to share the information implicitly. This tends to cause knowledge gaps to appear between the micro and macro level of the process

The main objective of the discussion is to bridge the gap between the scientists and the engineers. With the help of the initial model, questions such as "why do you think it happen", "how do you think it happened" and "what are the underlying science behind" will be posted to the stakeholders. They will be required to explain their belief explicitly and to support them with scientific evidences. This approach also enables all knowledge of the system to be shared explicitly and all the stakeholders share the same common knowledge and beliefs of the system.

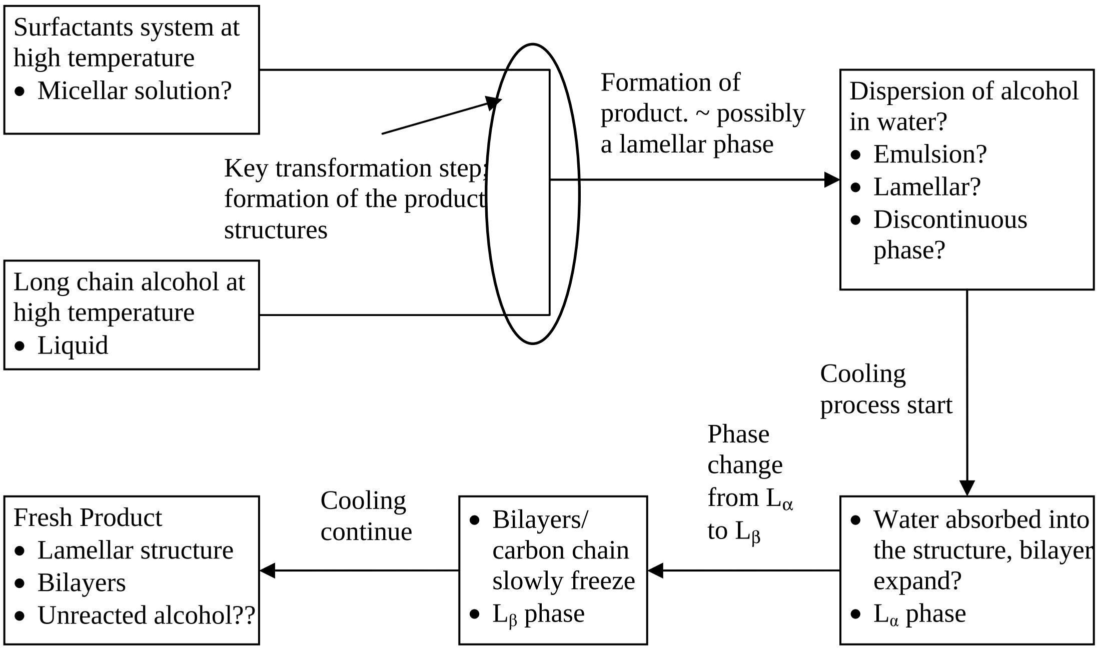

Figure 3 Initial version of the transformation map based on available information which identifies the key transformations which the materials experience and the states of the materials

2.4.1. Proposed Formation Pathway of Structure

During the "Question the Expert" several models for the process were developed and agreed upon. One of the models developed during the discussion is the formation pathway of the system structure. Figure 4 shows a schematic of the proposed formation pathway. The surfactants solution existed as a micellar solution with a mixture of micelles and monomer molecules. Upon the addition of the fatty alcohol, the monomers would "react" with fatty alcohol on the surface to form the structure. After this the monomers would diffuse through the structures to "react" with the rest of the fatty alcohol.

2.5. Specifically Designed Experiment – Penetration Experiment

Experiments were specifically designed to test certain parts of the pragmatic model. Three possible conclusions can be concluded from such experiments; the model proposed is credible, the model proposed is not credible and new phenomena occur previously not known to exist in the system.

A penetration experiment was designed and performed to test the credibility of the proposed formation pathway of the structure and to observe the behaviour of the system during the key transformation step. The two fluids involved were contacted on a microscope slide on a heated stage under similar manufacturing conditions under microscope. The contact interface between the fatty alcohol and the surfactant solution was observed.

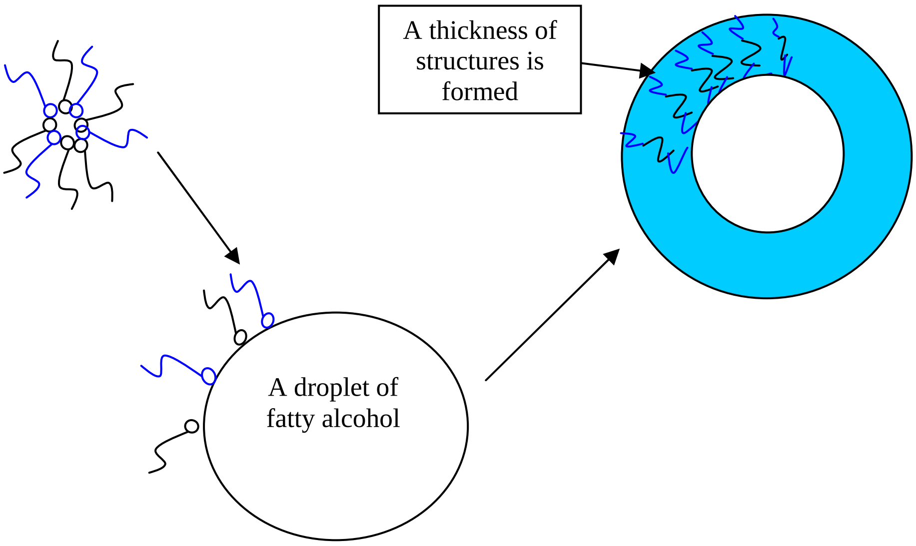

Figure 4 Proposed formation pathway of the formation of the system structures

Figure 5 shows the observation made from the penetration experiment. As soon as the surfactants solution came into contact with the fatty alcohol, it instantaneously “reacted” with the fatty alcohol. Throughout the experiment, the surfactants solution can be seen to be diffusing through the interface to “react” with the fresh fatty alcohol.

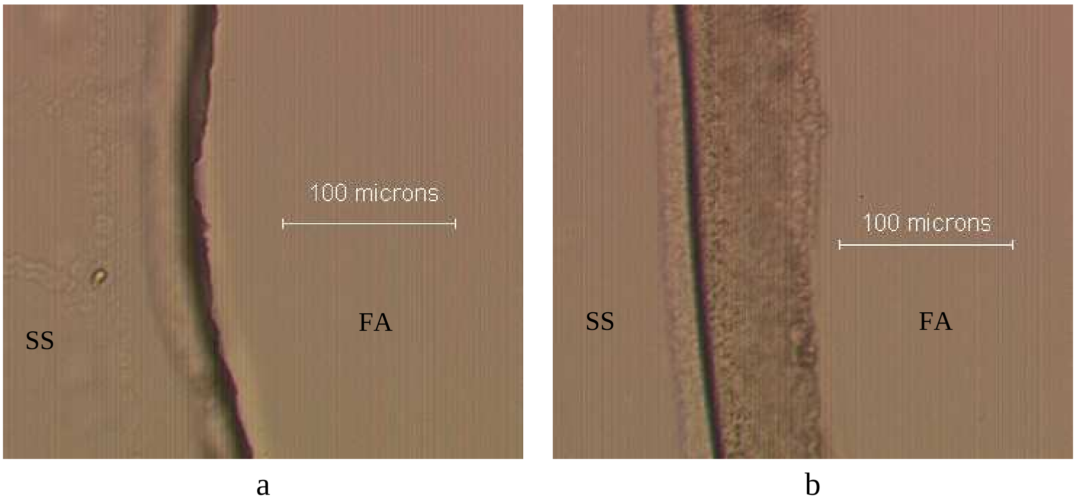

Figure 5 Pictures from the penetration experiments (a) when the fatty alcohol came into contact with the surfactant solution (b) the interface after 150s, where FA is the fatty alcohol side and SS is the surfactants solution side

Figure 6 shows the measured growth rate of a specific point along interface with respect to time. The growth rate appeared to increase at a rate of $\sqrt{t}$, which is consistent with a typical diffusion-limited process.

More importantly, observation indicated that the instantaneous "reaction" with the fatty alcohol is a phenomenon called "spontaneous emulsification" (Davies *et al.*, 1957). This is a new finding as there was no previous knowledge that such phenomenon existed in the system during the manufacturing process and could be an important finding.

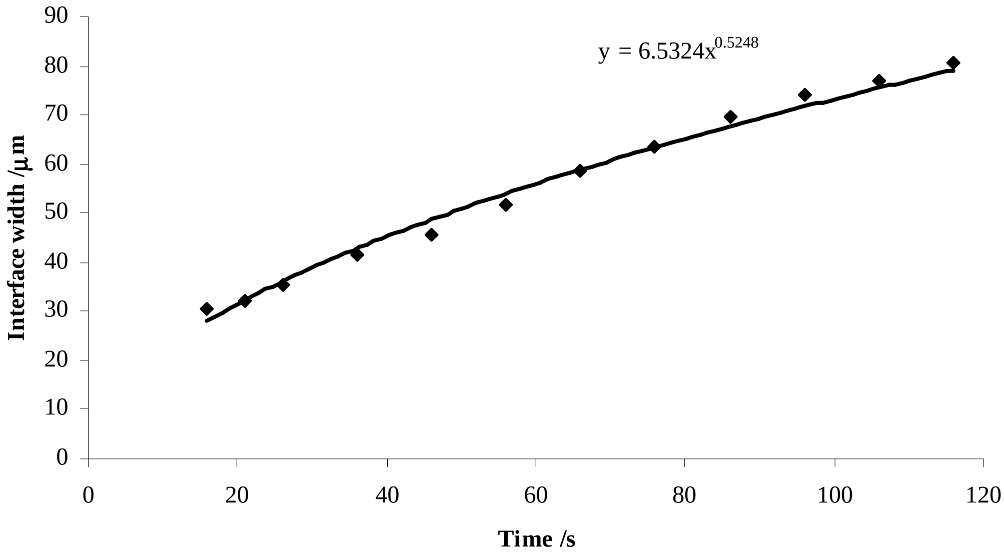

Figure 6 Growth rate of the interface when fatty alcohol contacted the surfactants solution

2.5.1. Proposed Limiting Feature of the System

During the addition of the fatty alcohol in the manufacturing process, the contact concentration between the fatty alcohol and the surfactants will not be constant. Figure 7 shows the contact concentration ratio between the fatty alcohol and surfactants during the manufacturing process, as experienced at the mixer downstream of the fatty alcohol addition point in the recirculation loop.

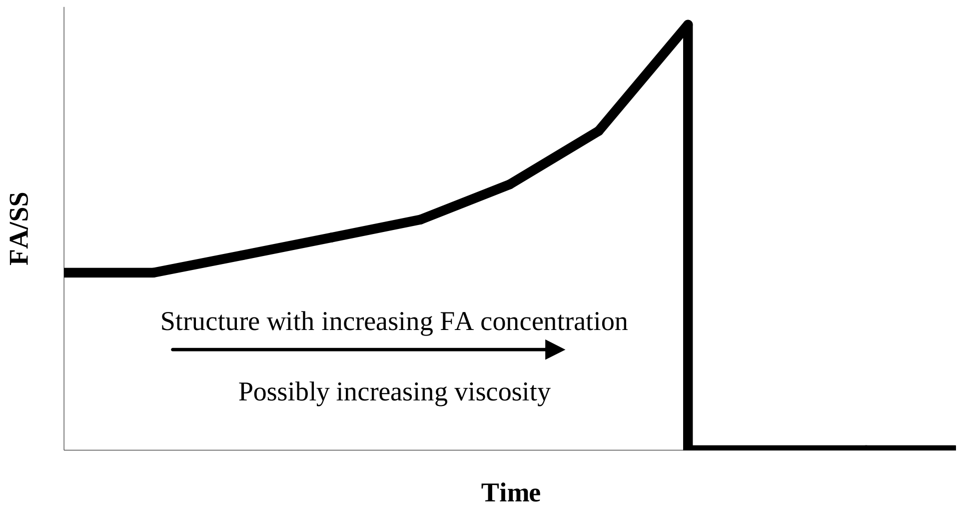

Figure 7 Fatty alcohol and surfactant concentration ratio against time during the addition of fatty alcohol (FA: fatty alcohol, SS: surfactant) for the standard manufacturing process

Given that spontaneous emulsification occurs in the system, there is a possibility that the product formed at different contact concentration would be slightly different in compositions and structure. This would create local inhomogeneity in the system in term of structure and composition and thus, a local viscosity profile within the product. The occurrence of spontaneous emulsification could be the limiting factor of the process.

There are two ways to mitigate the impact of spontaneous emulsification; the first is to have a mixing time faster than the occurrence of the phenomenon and the second is to ensure that the fatty alcohol and the surfactants solution contact each other at a constant concentration by changing the process configuration.

2.6. Specifically Designed Experiment - Modified Manufacturing Process

The batch process was modified to a semi-batch one whereby the contact concentration ratio between the fatty alcohol and surfactants solution can be maintain at a constant value. Figure 8 shows a schematic of the modified manufacturing process. The contact concentration ratio can be controlled via the flow rate of the materials.

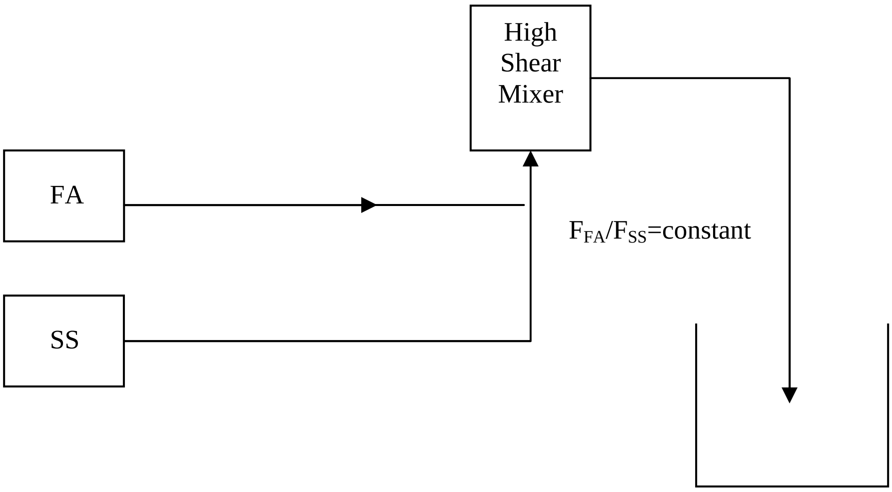

Figure 8 Schematic of the modified manufacturing process, F_{FA} is the flow rate of fatty alcohol and F_{SS} is the flow rate of surfactants solution

Four experiments were performed using different contact concentration ratio. In order for the overall composition to be the same, the required amounts of fatty alcohol or surfactants solution were added back into the system. Figure 9 shows the products viscosity manufactured at different contact concentration. Despite having the same overall composition, the product viscosity varied by factor of two just by changing the contact concentration ratio. This agreed with the proposal that the contact concentration is an important and possibly a limiting feature of the system.

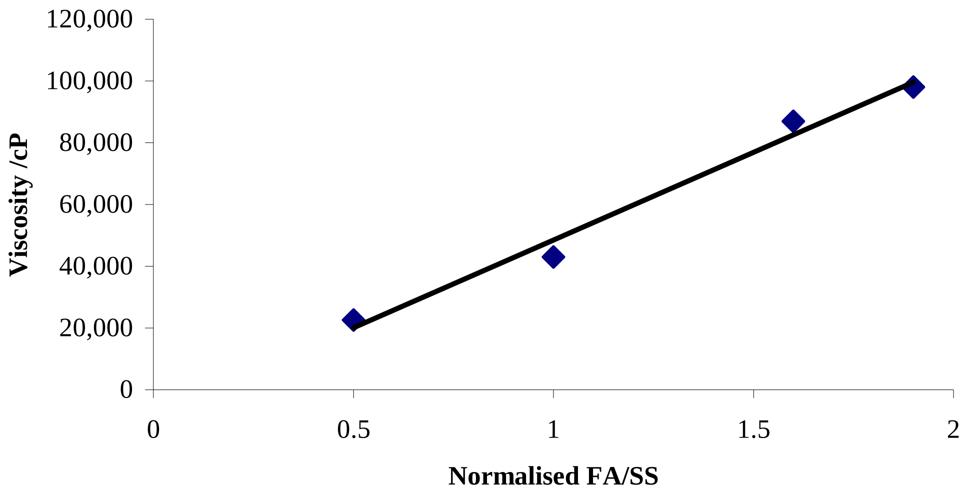

Figure 9 Products viscosity manufactured at four different contact concentration ratios

3. Conclusions

By adopting a mixture of pragmatic modelling and specifically designed experiments to probe the model, we were able to identify spontaneous emulsification occurs during the manufacturing process which was previously not known to exist in the system. By specifically designing a new semi-batch process, we were able to show that the contact concentration ratio is a crucial and possibly a limiting feature of the process.

References

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