A SOLIDS PRODUCT ENGINEERING DESIGN PROJECT

DHERMESH V. PATEL, AGBA D. SALMAN, MARTIN J. PITT, M.J. HOUNSLOW, I HAYATI*
The University of Sheffield • Sheffield S1 3JD, United Kingdom

The design project forms an integral part of an undergraduate degree in chemical engineering accredited by the Institution of Chemical Engineers[1] and the Institute of Energy. It is a four-year program in which a major design project contributes one-quarter of the credits in the third year. Usually, different projects (supervised by an academic staff member) are assigned to groups of 3 to 6 students. The groups have a period of one year to work through a typical process industry problem. For assessment purposes, the students make a verbal and visual presentation to staff and peers in the first semester and prepare a joint poster and a detailed individual design dissertation in the second.

The project provides a necessary understanding of process design of unit operations such as separators, distillation columns, heat exchangers, and other process components as well as bringing together other elements of the degree course, such as thermodynamics, transport phenomena, and process safety. In addition to supplementing the technical skills learned in lectures, the project develops transferable skills such as communication, organization, and team-working.

Traditionally, the design project has been geared toward designing a theoretical process for manufacture of a commodity chemical that dominated the chemical industry during the twentieth century, such as cumene or ethanol. It is relatively unusual for projects to involve much solid processing, despite its importance in industry.[2-4] This is in part because of the intrinsic difficulty and in part because data and design procedures are less readily available. In addition, student projects normally use purity as the main or sole measure of the product's quality. For many solid products, however, the particle size distribution, flowability, and functionality in use may be equally or more important.

In the past, chemical engineers have designed processes but have largely left product specification to others. In recent years, however, it has been suggested that they should be actively involved in product design, particularly for solids.[5,6] Courses and theoretical projects on product design now exist in some European and North American universities,[7] and there is now an undergraduate textbook.[8]

This project was restricted to MEng students. They are first-degree students who have achieved a higher minimum standard (55% instead of 40%) in earlier courses and who complete an additional year of study compared with BEng students. The project was offered to allow such capable and well-motivated students to actively engage in the design process for a real industrial product. Before starting the project, they had completed two years of laboratories and a mini-project. In the current scheme, students study particle science in the second year and particle processes in the third year.

A number of universities include experimental work as part of the MEng degree scheme, but they are generally research

Dhermesh V. Patel *graduated with a first class degree in Chemical Process Engineering and Fuel Technology. While at Sheffield University he received the British Coke Research Association Prize and the Vacation Work Prize, and was awarded the Mappin Medal for his outstanding final year performance.*
Agba Salman *is a Chartered Physicist, formerly with the Open University and now a Lecturer in Chemical and Process Engineering at the University of Sheffield. Both his teaching and research are concerned with particle technology. He was the principal supervisor for the project.*
Martin J. Pitt *is a Chartered Chemical Engineer, with industrial and academic experience, currently the Co-ordinator of Design Teaching in Chemical and Process Engineering at the University of Sheffield.*
Michael Hounslow *has a doctorate in chemical engineering from the University of Adelaide. He is currently Head of Department and leader of the Particle Products research group in Chemical and Process Engineering at the University of Sheffield.*
Igan Hayati *studied chemical engineering at Leeds University. He then joined the Interface Science group at Imperial College and obtained his Ph.D. in 1985. He worked for ICI, Paints Division for three years and then joined the research Department at Borax in 1990. He is now responsible for new product development and new applications at Borax Europe.*

---

* *Borax Europe Ltd, Guildford GU2 8XG, United Kingdom*

projects (albeit of an applied and often interesting nature). The project described here is unusual in that it uses laboratory measurements as part of a design exercise and uses real industrial materials.

BACKGROUND

Boron is one of seven essential micronutrients required for normal growth and fruiting of most agricultural crops. Soil testing and plant tissue analyses have detected that of the essential micronutrients, boron is usually the most deficient in crops. Therefore, annual applications of boron are required for high yields and improved quality and to offset losses from crop removal and leaching. Plants, depending on soil type, management level, and method of application, require only small quantities of boron—around 0.2 to 4 kg per hectare per year.[9] Also, since borates are toxic to wood-boring insects but beneficial for plant growth, they are used as a wood preservative.

Boron is conventionally supplied in the form of granular borate compositions. In applications that require spraying, such as in agriculture, aqueous borate suspensions are prepared by heating, dissolving, and rapid cooling of the granules, incurring practical difficulties and additional costs. These suspensions are not self-structured and thus require the addition of a thickening agent to maintain the stability of the suspension.

Borax, a global supplier of borates, now has a patented self-structured aqueous borate suspension[10] that does not require a thickening agent to suspend the particles. Sodium pentaborate pentahydrate ($NaB_5O_8.5H_2O$) is formed by reacting boric acid (H_3BO_3) with borax pentahydrate ($Na_2B_4O_7.5H_2O$) in the presence of water.

$$6H_3BO_3 + Na_2B_4O_7.5H_2O \rightarrow 2NaB_5O_8.5H_2O + 4H_2O$$

The product conveniently referred to as "borate cream" is sodium pentaborate pentahydrate and is, as yet, the only stable aqueous borate suspension found. As a consequence there is limited knowledge of the intermediate steps involved in its production. The suspension has a high solid content of 46 wt% and a 10% boron content. The reason the suspension is self-structured is believed to be caused by the weak attraction of the particles by van der Waals forces.

The self-structured suspension has many advantages over the previously prepared borate suspensions. Not only is the suspension physically stable, but it is also pourable (unlike the stiff compositions previously produced). The suspension can then be readily diluted in water for application, providing greater convenience for the consumers.

The demand for the "borate cream" in plant nutrition and wood preservation is expected to increase in the future. Other applications for the cream could also be discovered due to the diverse properties it exhibits.

To date, the cream has been entirely produced in a batch process. Thus, Borax is collaborating with the University of Sheffield to establish the feasibility of increasing production by implementing a continuous process.

PROJECT WORK

A group of four third-year chemical engineering students were given the task of meeting the project objective. The group first arranged a meeting with a Borax representative to discuss the requirements of the plant design and to obtain a more complete description of the current process. The company recommended features of the plant layout, sizing of equipment, and a production of 50,000 tonnes per year of 10% boron content cream. Product stability was critical, so it was necessary to investigate additives to prevent syneresis (separation) of the cream.

From this data, a preliminary overview of the process was devised. The process could be divided into four sections: silos, premixing and storage, solid conveying, and reactors.

In order to design and select the appropriate equipment for the handling and storage of these components, essential data on the nature of the bulk solids and water would have to be determined. The physical and chemical properties of water could be readily obtained from published data, but due to the originality of the process, relevant design data for the solid additives (such as specific densities and particle sizes) were less readily available. Therefore, these parameters were measured experimentally by the students to give the properties directly for the conditions that would be encountered in the design. In addition, the cream was produced by using the current batch procedure to give the students further insight into the process. This data could then be used to design the individual components of the continuous production plant.

Finally, a collective consideration of the design, economics, and safety and environmental aspects of the final proposed design was performed. The following paragraphs describe the work of the students in terms of both experimental and design work.

EXPERIMENTAL WORK

The experimental work consisted of three major constituents: measuring the physical properties of the reactants and products, determining the formation characteristics, and measuring the viscosity of the final cream.

Properties of the Reactants • The physical properties of the components that are vital in the design of the mixers, storage silos, and transportation system are the angle of repose, the bulk density, and the particle size, shape, density, and porosity.

The repose angle is required in the design of the storage silos and belt conveyors. It is the angle the particles make with a flat surface when a quantity of solid is allowed to form a heap. A standardized test procedure was used to give the "poured" angle of repose for a given bulk solid.

In powder-handling systems, particle size is a key parameter in design calculations. There are a variety of particle-size-measurement techniques available in the department, but due to the smaller particles present in the powder distributions, the preferred measurement techniques were the laser-diffraction technique (LDT) and sieving. The general operating principle of LDT is that the angle of diffraction of a beam of light passing through the particles depends on the wavelength of the light and the size of the particles. We used the apparatus located in the department to give the particle size for powders within the measurement range of 4.5 to 875 μm, with sieving used for all other circumstances. The frequency distribution obtained for additive "A" is shown in Figure 1.

The shape of particles can have a significant bearing on the packing and flow behavior of the bulk solid. Samples of boric acid and borax pentahydrate were analyzed underneath a low-powered microscope with suitable photographic attachments to determine the particle shapes. Micrographs of boric acid and borax pentahydrate are given in Figure 2.

It was evident from the micrographs that the particles were non-spherical and that some were even agglomerates. Hence, since nonspherical particles can affect the flow behavior and equipment wear, the particle's shape was taken into account in the silo and conveying design.

Also, the particle density, bulk density, and porosity of the powders were measured using standard tests. All the measurements performed on the four bulk solids are summarized in Table 1.

<u>Formation of the 'cream'</u> • The quality of the cream generated is highly dependent on three conditions of formation: agitation rate, concentration of solid components, and temperature of operation.

The cream was produced under batch conditions in the laboratory to determine the optimum conditions to produce it in a continuous process, taking into account production, economic, and safety factors.

Initially, the cream was produced without any anti-settling agents,

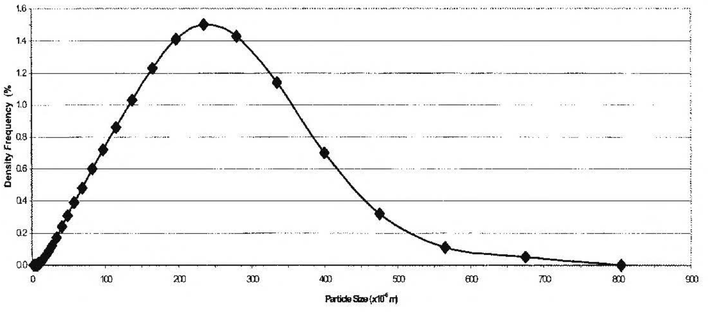
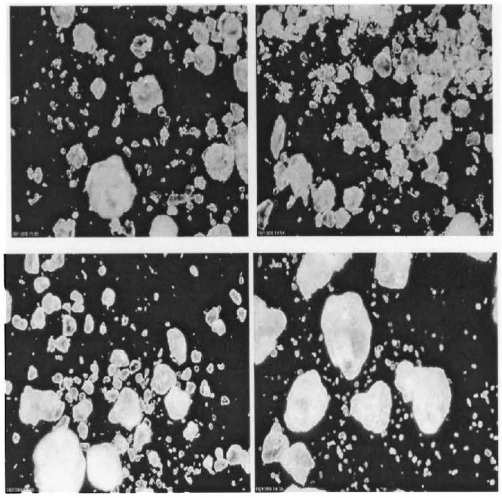


Figure 1. *Frequency particle size distribution for additive 'A'.*



a) Borax Pentahydrate

b) Boric acid

Figure 2. *Micrographs of reagents: a) borax pentahydrate b) boric acid.*

Property		Boric acid	Borax pentahydrate	Additive 'B'	Additive 'A'
Angle of Repose		34.5°	36.5°	32.0°	51.0°
Particle Size Parameters (μm)	x_{16}	142.80	190	2.70	77.2
	x_{50}	255.75	450	8.65	182.5
	x_{84}	444.70	860	32.50	316.8
	σ	1.76	2.13	3.49	2.03
Elongation		1.25	1.20	-	-
Bulk Density (kg/m^3)		950	1060	-	-
Particle Density (kg/m^3)		1520	2420	720	1760
Porosity		0.375	0.562	-	-

Table 1. *Summary of physical measurements of the bulk solids.*

but syneresis occurred when the suspension was allowed to stand for long periods, resulting in two distinct layers: an aqueous and a solid phase. Although mixing could readily restore homogeneity, this would greatly affect large-scale production. We found that adding two anti-settling agents to the water prior to adding the reagents minimized separation. Hence, later cream productions involved an additional premixing stage to hydrate the anti-settling agents prior to adding the reagents.

Understanding the mechanism of the cream formation is essential since it can allow possible improvements to the manufacturing process and could lead to the production of other borate suspensions. Therefore, samples taken at various intervals during the production of the cream were analyzed (in relation to temperature and pH readings) to determine the stages of production. It is known that mechanism of the formation of crystals involves two major phases: dissolution and nucleation.

A general temperature trend during the reaction phase was a U-shaped curve, as shown in Figure 3. Upon addition of the reactants, the temperature dropped sharply, resulting in a minimum temperature after approximately 20 minutes. We found that this period corresponded to the dissolution of the solid reagents in water via an endothermic process.

The temperature increased steadily between 20 and 60 minutes of reacting, but remained below the temperature before the addition of the reactants. This temperature rise was found to be due to the commencement of nucleation, which is an exothermic process.

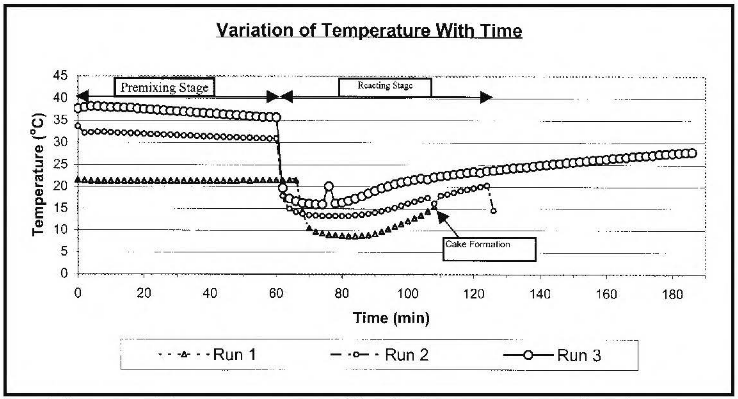


Figure 3. *Temperature profile during cream production at varying initial temperatures.*

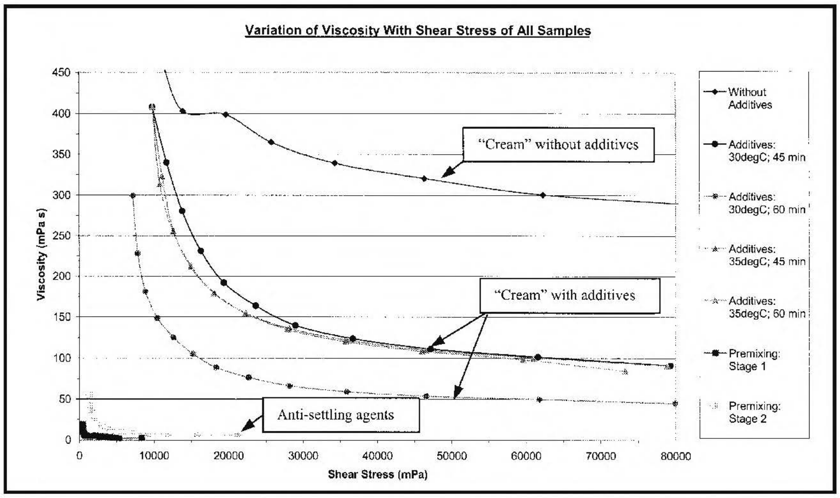


Figure 4. *Variation of viscosity with shear stress for the cream produced with and without additives and the anti-settling agents.*

The experimental data allowed us to determine the effect of conditions on dissolution and nucleation (and hence the product quality) and were used to design the reactor stage under the optimum conditions for the desired product quality.

Viscosity Measurements • The viscosity is a fundamental fluid property that is necessary to predict the manner in which a fluid will react to applied forces such as pumping. Since the cream is non-Newtonian and exhibits complex flow behavior such as separation, the viscosity had to be determined experimentally for the conditions that would be encountered.

We measured the viscosities of the samples in a Rheomat 115 rotational viscometer located in the department. Its coaxial measuring system operates according to the Searle principle. The control instrument enables the rotational speed to be varied and the torque readout to be recorded. The shear rate and shear stress are determined from the rotational speed of the bob and the braking torque indication, respectively, allowing the rheogram to be plotted.

The viscosity of the cream should be as low as possible to allow ease of handling and to allow the cream to be dispersed in water during application. The variation of viscosity with shear stress for the cream produced with and without the additives and the anti-settling agents is shown in Figure 4. The experimental data suggest that for the cream produced with the anti-settling agents, high mixing time during the reaction stage and high temperature tend to give lower viscosity. The conclusions from this investigation were again incorporated into the process design.

DESIGN WORK

Design of the proposed process was divided into two main sections: design of the individual components of the process and overall process design.

Component Design • The proposed process was divided into four distinct phases: containment of solids, solid conveying, premixing, and reaction. A flow diagram of the proposed continuous plant is given in Figure 5 (next page). The process features a storage silo for each of the solid components. The two solid reagents have storage and feed silos to contain the large quantities of materials required. The silo design included material properties determined from shear testing using a Jenike shear cell.[11]

Pneumatic conveyors transport the material from storage to the feed silos, with cyclones positioned adjacent to the feed silos to separate the particles from the air stream. The solid conveying design included evaluation of the required air flow rate to give steady operation, design of a cyclone separator, and specification of a suitable air mover and rotary valve to discharge the cyclone and act as an air lock. As there are many system specifications that could be used, evaluation of the best design was performed. All the solid components are gravity fed from the feed silos to the first CSTR.

Since the two anti-settling agents comprise only a small part of the final cream, they are only stored in feed silos that are refilled manually. The anti-settling agents are hydrated in two batch premixers working alternately. The components need hydrating for a specific time of 1 hour, so the process has to be performed in batches. A continuous feed to the reactors is obtained by allowing the premixers to work alternately.

The reaction occurs in four reactors in a series arrangement. By increasing the number of reactors in operation, the process shifts from a continuous to a batch process, thus increasing the likelihood of complete reaction. Due to economic factors, however, four CSTRs in series were chosen. The reactors were designed by scaling up the laboratory data so that dissolution of the solid reagents in water occurs in the first CSTR and nucleation in the subsequent reactors.

The finished cream product is then stored in two large tanks prior to transportation to consumers.

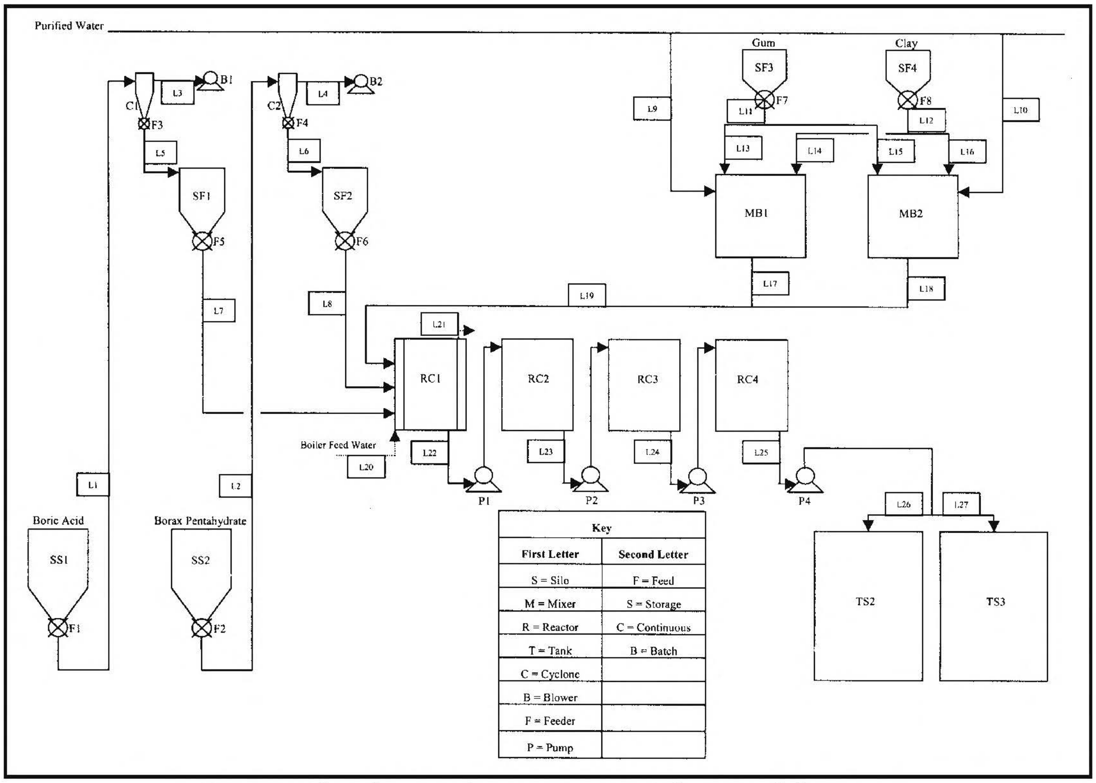


Figure 5. Flow diagram for continuous production of the cream.

Overall Process • A collective consideration of the design, economics and safety and environmental aspects of the final proposed design was performed. A detailed cost analysis was carried out on the final plant design. The figures derived by the students were given and the capital cost of the plant was estimated by using the factorial method. The purchase cost of equipment was obtained from quotations, when possible, to increase accuracy of the analysis.

The plant should be inherently safe since the process is enclosed and safe operation is inherent in the nature of the process. The solid reagents, the additives, and the 'borate cream' are not flammable, combustible, or explosive. Additive 'A' is combustible, but since it comprises only 0.1% of the cream, the danger is likely to be minor. Dust exposure can be controlled by a combining engineering and process control to prevent airborne dust concentrations. Basic safety and fire preventative measures were included in the design. Overall, the process should cause negligible damage under foreseeable circumstances.

A hazard and operability study (HAZOP)[12]—a systematic, critical examination of the operability of a process—was performed to indicate potential hazards that could arise from deviations from the intended design. A partial HAZOP Result Sheet is shown in Table 2. Any additional safety features from the analysis were included in the final P&I diagram. The unit P&I diagram for the premixing stage is given in Figure 6.

CONCLUSIONS

Borax expressed its delight on a vital piece of work that would otherwise have been performed by the company. The exercise showed that students are capable of taking an active role in an industrial design, not as part of an industrial year but as a major assessed project carried out at the university. The use of experimental measurements to define product performance rather than simply to collect property data was a valuable experience.

Student feedback indicates design projects tend to develop teamwork, presentation, and technical skills. In this particular case, the students also felt that obtaining experimental data on the product properties and producing the cream in the laboratory gave greater insight into the fundamental aspects of the process and provided a better means to meet the process objectives. Another beneficial aspect was dealing with company representatives and actual components rather than just theoretical data.

TABLE 2
Partial HAZOP Result Sheet

No flow	Blockage in line 9/10 Valve V3/V4 failure PLC faulty	Hydration does not occur	Put flow indicator in line 9/10
More flow	Valve V3/V4 failure	Insufficient hydration PLC faulty	Put weight control in premixers to detect quantity of material
Less flow	Valve V3/V4 failure	Insufficient hydration PLC faulty	Put weight control in premixers to detect quantity of material
Early flow	Timer faulty on PLC for V3/V4	Feed may enter in previous batch	Regular checks on timer on PLC
Late flow	Timer faulty on PLC for V3/V4	Feed may enter in next batch	Regular checks on timer on PLC

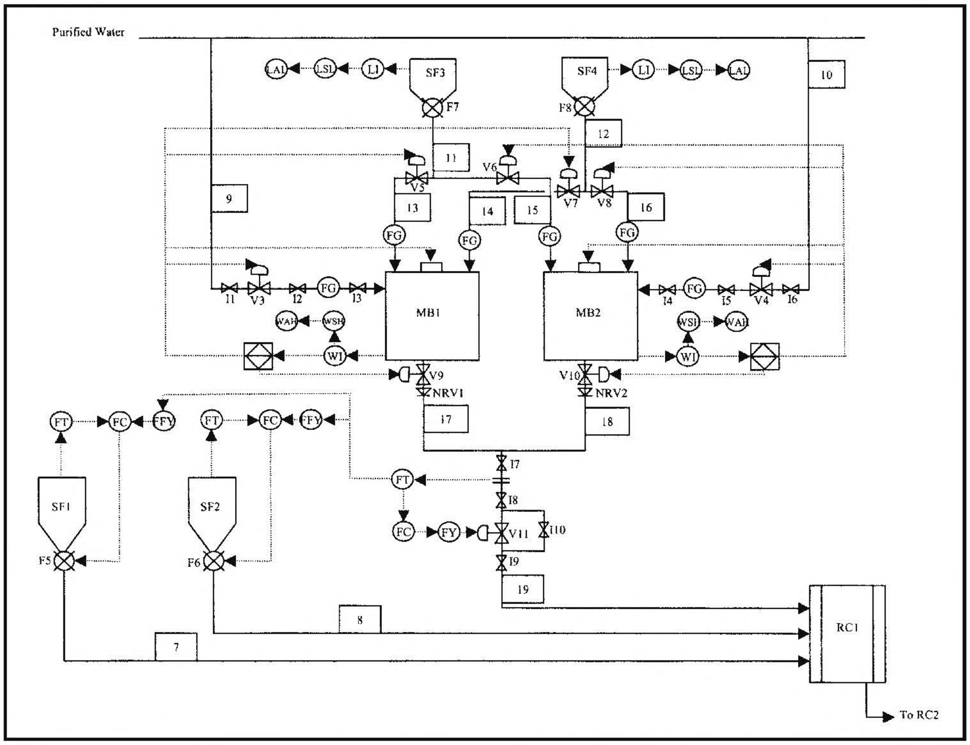


Figure 6. Unit piping and instrumentation diagram.

For traditional design projects, the problem with undergraduates getting experimental data is that materials in many traditional processes are toxic and the conditions involve high temperatures and pressures. In comparison, the materials involved in this process were relatively benign and the conditions were moderate. Many other industrial processes involving solids would also fall into this category. In the future, we hope to have more design projects that combine laboratory work with engineering design, preferably based on actual problems—collaboration that benefits students and industry alike.

ACKNOWLEDGMENT

We would like to acknowledge the work of group members Jenny Richardson, Richard Heath, and Andrew Brown. Finally, we are grateful to Borax for allocating the project to the department and providing essential information and assistance.

REFERENCES

1. Institution of Chemical Engineers, "Accreditation of University Chemical Engineering Courses," *IChemE,* Rugby, UK (2001)
2. Bridgwater, J., "Particle Technology," *Chem. Eng. Sci.,* **50**(24), 4081 (1995)
3. Nelson, R.D., and R. Davies, "Industrial Perspectives on Teaching Particle Technology," *Chem. Eng. Ed.,* **32**(2), 98 (1998)
4. Chase, G.G., and K. Jacob, "Undergraduate Teaching in Solids Processing and Particle Technology," *Chem. Eng. Ed.,* **32**(2), 118 (1998)
5. Villadsen, J., "Putting Structure into Chemical Engineering," *Chem. Eng. Sci.,* **52**(17), 2857 (1997)
6. Cussler, E.L., "Do Changes in the Chemical Industry Imply Changes in Curriculum?" *Chem. Eng. Ed.,* **33**(1), 12 (1999)
7. Shaeiwitz, J.A., and R. Turton, "Chemical Product Design," *Chem. Eng. Ed.,* **35**(4), 280 (2001)
8. Cussler, E.L., and G.D. Moggridge, *Chemical Product Design,* Cambridge University Press (2001)
9. Borax Company website (2001): <http://borax.com>
10. Hayati, I., Aqueous Borate-Containing Compositions and Their Preparation, Patent # Wq 99/20565
11. ASTM: D6128-00 Standard Test Method for Shear Testing of Bulk Solids Using the Jenike Shear Cell
12. Kletz, T., *Hazop and Hazan,* 4th ed., IChemE, Rugby, U.K. (also AIChE) (1999) ❐