

# **INDUSTRIAL APPLICATIONS OF SPINNING CONE COLUMN TECHNOLOGY: A REVIEW**

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## **ABSTRACT**

The Spinning Cone Column (SCC) is a mechanically assisted gas-liquid contacting device which has been successfully applied to a broad range of distillation processes in the food and beverage industries. SCC technology is now being adopted for a variety of specialised tasks in the chemical and process industries.

In this review, the technology is described and its operating principles explained. An account of the evolution of the technology, from isotope enrichment to the separation of aroma compounds from food and beverage streams, is presented.

The efficacy of SCC technology in food and beverage processing has prompted the investigation of potential uses with non-food materials. The development of non-food applications is now proceeding rapidly: removal of volatile organic compounds (VOCs) from highly viscous polymeric suspensions, and the continuous removal of volatile fermentation by-products are described to illustrate the growth of this powerful technology.

## **INTRODUCTION**

The spinning cone column (SCC) is a distillation device now used in the food industry for the separation of volatile components from liquids and slurries [1, 2]. It consists of a vertical succession of alternate rotating and stationary cones. Liquid flows as a thin film down the stationary cone, drains into the base of the rotating cone whence it moves upward and outward on the surface of the rotating cone, again as a thin film, by the action of centrifugal force. Gas flows up the column countercurrent to the flow of liquid.

SCCs have lower gas pressure drop and lower liquid hold-up than do traditional plate and packed columns, hence their suitability for distillation of thermally sensitive foodstuffs. Established commercial applications of the technology include alcohol

adjustment of wines, and the recovery of volatile flavours from various food and beverage streams, including coffee, tea, citrus products and other fruits and vegetables. The nature of the fluid flow pattern obtaining in the SCC renders it particularly suitable for the processing of highly viscous liquids and slurries, materials which cannot be handled by most traditional columns.

## THE EVOLUTION OF SPINNING CONE COLUMN TECHNOLOGY

### Origins

The first published references to the spinning cone column appear in 1936 in two papers by Pegram, Urey and Huffman [3, 4]. A year later Huffman and Urey [5] gave the first detailed description of a spinning cone column, and presented the results of trials on the separation of isotopes of oxygen in water; the object was to produce "sufficiently large quantities of the heavier isotopes of this element for future physical, chemical and biological researches."

It had been shown a few years before this that heavy oxygen water ( $\text{H}_2\text{O}^{18}$ ), is about 0.3% less volatile than normal water ( $\text{H}_2\text{O}^{16}$ ). Efforts were made to exploit this difference in vapour pressure to separate the two materials by distillation. It was recognised, however, that existing designs of distillation column did not offer the performance required for such a demanding separation, hence the attention paid to alternative configurations.

Pegram, Urey and Huffman observed a wide variation in mass transfer performance (stage efficiencies between 25 and 100%). Although their work did not extend to systematic investigation of such variations, it did represent the crucial first step in the development of the technology; the concept of the SCC (internal layout and principle of operation) was established and, most importantly, the device was demonstrated to function in the manner envisaged by its originators.

The next major investigation of the SCC was reported in 1939 by Mair and Willingham [6], who were concerned with high-efficiency laboratory distillation columns. They looked at the effects on performance of different types of rotating element and internal geometry and, as a result, established that the basic (two-cone) configuration gave the best performance, at least with respect to mass transfer (stage efficiencies between 50 and 80%). An important implication of this aspect of their work was that the contact between gas and liquid droplets, maximised in the case where perforated baskets were used as rotating elements, did not appear to be the pre-eminent mode of mass transfer between the phases.

The publication of research findings on the SCC lapsed after the early (pre-war) American work until 1963, when Ziolkowski *et al.* [7] reported studies on the operational characteristics of a spinning cone column used for the separation of a benzene-carbon tetrachloride mixture.

The work of Ziolkowski *et al.* was similar to the pre-war American work in that all results were obtained (with the SCC running) under total reflux. It was the first study in which gas pressure drop data were presented and in which the influence of rotor speed, on

both mass transfer performance and gas pressure drop, was considered. Separation efficiency increased with rotor speed at all throughputs; at a given rotor speed separation efficiency tended to fall as throughput was increased (in a similar manner to that observed in packed columns).

### **The Use of the SCC for Separation of Food Volatiles**

In the 1960s, Dr D.J. Casimir of the CSIRO Division of Food Research was engaged in work on the processing of fruit juices, particularly products derived from passionfruit. He was particularly concerned to find ways of producing concentrated (evaporated) passionfruit juice which retained the distinctive and highly desirable aroma and flavour of the raw material.

The basis of the problem that Casimir addressed was as follows: fruit juices are almost always evaporated and in the course of the removal of up to 85% of the original water content, a significant fraction of the juice's volatile components will be removed as well. These volatile components are critical to the sensory character and perceived quality of the juice, and so must somehow be separated from the water and restored to the concentrate or to the reconstituted juice if the flavour and aroma of the final product is not to be substantially degraded.

Various evaporator-based systems have been developed for the recovery of volatile components from fruit juices, typically comprising a single-stage flash evaporation prior to the main evaporation step, and/or rectification of condensate from one or more effects of the evaporator.

Casimir's work on the evaporation of passionfruit juice led him to conclude that such systems would never provide an entirely satisfactory solution to the problem of volatile degradation and loss; simple systems inevitably gave crude results, and the more refined systems rapidly became highly complex and expensive. Instead, he took the view that the separation of volatiles was best effected prior to the evaporation step and that this would require a counter-current gas-liquid contacting device which offered a high mass transfer capacity, so allowing acceptable recovery of even the least volatile components, but which did not require operating conditions which would bring about damage to the product. On this basis the requirements for a device to be used for separation of heat labile volatile materials by distillation were [8]:

- (a) countercurrent gas and liquid flows
- (b) large interfacial gas-liquid contacting area
- (c) high turbulence within gas and liquid phases
- (d) little or no entrainment
- (e) low liquid hold-up volume
- (f) low pressure drop

Casimir concluded that of all the possible configurations the SCC best met his stated criteria.

With the exception of size and geometry, the SCCs used by Mair and Willingham, and by Ziolkowski *et al.*, were essentially the same as the devices used by Huffman and Urey. Casimir's principal change to this basic design was the addition of radial fins to the undersides of the rotating cones. This effectively turned each spinning cone into a

centrifugal impeller, and the whole column a multi-stage centrifugal fan; this fan action could be used to control pressure drop across the column. In addition, the rotatory motion imparted to the vapour not only limited entrainment of liquid in the vapour stream but also increased agitation in the vapour phase, thereby enhancing mass transfer within it.

Whereas the bulk of earlier work on the SCC was concerned almost entirely with mass transfer performance, Casimir cited a number of characteristics, in addition to high mass transfer efficiency, which made the SCC likely to perform well in food applications: low pressure drop, low liquid hold-up and short residence time. These aspects of SCC performance were important in food applications because of the typically acute heat lability of volatile components of foods.

Menzi [9, 10] extended the work of Casimir by comparing various aspects of the performance of a spinning cone column with those of a similarly sized bubble cap column. Menzi's SCC had, like Casimir's, radial fins attached to the undersides of the spinning cones. Menzi presented data on liquid residence time distributions which showed the pronounced difference between the SCC and a similarly sized bubble-cap column in this aspect of their performance. These data confirmed Casimir's findings with respect to residence time and supported his view that the SCC offers significant advantages in this regard in the processing of heat-labile materials.

In 1990, a collaborative programme of research into the engineering aspects of SCC technology, involving Casimir and S.J. Sykes of CSIRO, and Professor R.G.H. Prince of the University of Sydney's Department of Chemical Engineering, was initiated. Up to this time, the SCC had been shown to offer advantages in the areas of mass transfer, pressure drop and liquid residence time. There were, however, no correlations of physical capacity limits and mass transfer performance which could be used for design. There followed a series of publications in which this lack of design data was addressed [11]. A key area of investigation in this programme was the prediction of flooding in the SCC. Flooding data obtained on SCCs of various sizes were satisfactorily correlated using a relationship of the same form as the Sherwood-Leva-Eckert correlation for packed columns. Also, mass transfer was shown to be sensibly constant over the operating range of the SCC, thus allowing design to be based on capacity considerations. Research on the SCC took a new turn in 1992 when the flow of liquid films on rotating conical surfaces became the focus for further work by Prince and his colleagues Associate Professor T.A.G. Langrish and Dr S. Makarytchev at the University of Sydney [12]. Their searching and comprehensive analysis of the SCC's fluid dynamics has prompted substantial and continuing effort directed at devising computer-based simulations of both liquid and gas flow in the column.

Work has continued over recent years to further investigate and optimise the stage efficiency of commercial Spinning Cone Column plants. Simplified approaches such as the McCabe-Thiele approximation have been used to interpret data generated from a two component model system composed of ethanol and water. The results of these studies have indicated that stage efficiencies in excess of 85% are achievable.

## FOOD APPLICATIONS DEVELOPMENT

In parallel with the technical developments described earlier in this paper, numerous commercial applications were developed over the period from 1986 to the present day. Early commercial trials between 1984 and 1986 were concerned with the removal of sulphur dioxide (used as a preservative) from grape juice. The SCC was found to be more efficient and to produce a better quality treated juice than the conventional bubble cap columns that had been widely used in the 1970s. During this time it was found that the SCC could also be used successfully to recover aroma and remove alcohol from wine. Wine treatment quickly became one of the major commercial applications for SCC technology, whereby the ratio of aroma to alcohol in wines could be managed in what is now generally termed 'alcohol adjustment'. The annual volume of wine processed in this way on the SCC in the USA alone has exceeded 30,000,000 litres per annum.

Early commercial units were also sold into the citrus and coffee industries. The first applications in the coffee industry were for the recovery of aroma from coffee extract; the aroma fraction was added back to improve the flavour of soluble coffee products. The early impact of the SCC technology in the soluble coffee process laid the foundation for it becoming widely adopted in the coffee industry throughout the 1990s. During this period the soluble and ready-to-drink coffee market grew significantly around the world, as did the sophistication and general quality of products being developed for the consumer.

The start of the 1990s also saw the development of a major application in the dairy industry. The removal of volatile off notes from cream used for butter making had been routine in the New Zealand dairy industry for many years prior to 1990. The viscosity of the cream and the requirement for hygienic process equipment, however, meant that relatively simple flash evaporation technology had been used as the standard technology. Trials with the SCC showed that not only could the SCC handle the increased viscosity of the cream but that the steam usage to remove the volatile compounds was reduced by 80% compared to the flash evaporation process. Moreover the ability to clean in place (CIP) effectively in an environment where hygienic operation is critical was demonstrated. SCC plants in the dairy industry were also required to be remotely operated and integrated into the rest of the process. This requirement led to the development of fully automated SCC plants; similar levels of automation have since been built into SCC plants destined for other sectors of the food industry.

Work first completed in the mid 1990s on recovering aroma from tea led to the conclusion that highly significant yield and quality improvements could be gained by processing tea slurry, as opposed to tea extract. The slurry would typically be prepared by adding 10% by weight of leaf tea to water (see Figure 1). Such a feed material could simply not be processed in conventional multistage columns. A similar approach was soon being investigated for processing coffee slurry and, as in the case of tea, the ability to process coffee slurries containing up to 20% suspended

solids facilitated the recovery of much 'fresher' (roast and ground) flavours than is possible from extract.



*Figure 1: A 10% slurry of green tea being discharged from a Model 500 SCC after stripping of volatiles. The flow rate was 300 L/h.*

Several areas of SCC technology have been substantially expanded during the development of slurry processing applications. These include specialised CIP techniques and systems for controlling the flow pattern of high solids feed materials inside the column. The need to maintain the homogeneity of slurries in the column, particularly at the feed point, has led to the design and exhaustive testing of new mechanical arrangements. As a result, the processing of tea and coffee slurries is now routine on purpose-built SCC systems. The recovery of high quality herb and spice volatiles continues to extend the principle of aroma recovery directly from solids presented to the SCC as a pumped slurry.

The ability of the SCC to handle highly viscous materials have been demonstrated in the course of processing particular fruit products such as single strength purees of mango and banana. Such products have viscosity in the range 2500cP to 4000cP at the processing temperatures used in the SCC. Figure 2 shows a commercial plant processing banana with an enlargement inset showing the view through the bottom sight glass. The processing of mango and banana has brought to light an additional benefit of steam stripping on the SCC: the reduction of dissolved oxygen from the product stream. Low oxygen levels limit the undesirable browning of the puree during subsequent aseptic filling and other packaging operations.



*Figure 2: A Flavourtech Model 10,000 SCC being used to process banana puree. Inset is an enlarged view of the sight glass in the discharge line, showing the puree being pumped out of the column. The flow rate was 3,500 L/h.*

## **DEVELOPMENT OF APPLICATIONS IN THE CHEMICAL AND PROCESS INDUSTRIES**

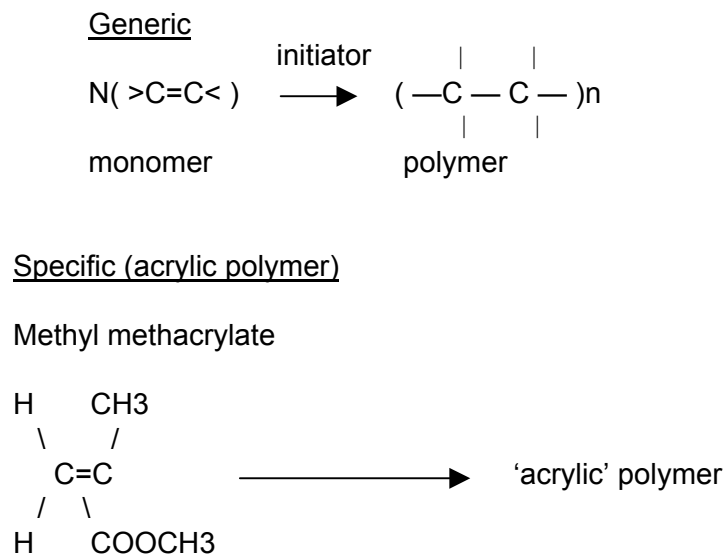
The SCC has been successfully applied as an efficient and highly flexible mass transfer device in the food sector. Major applications in the food sector have developed in those areas where the particular physical and operational characteristics of SCC technology could be used to bring about significant improvements in process efficiency and product quality. These same features are now being used to tackle some demanding applications in the Chemical and Process Industries.

### **Paint Products**

An example of such an application is the removal/reduction of volatile organic compounds (VOCs) from polymer latices used in the manufacture of paint products.

The need to remove VOCs from paint products is driven by several factors. Firstly, users, whether commercial or domestic, of the final paint products prefer to avoid creating an unpleasant environment when the paint product is applied. Secondly, in some product sectors legal limits on residual VOCs are being introduced or tightened further in order to limit exposure to compounds that may pose a health risk on lengthy exposure. Thirdly, if the compounds can be removed in a non-destructive way there is an opportunity to recycle them back into the process and reduce monomer usage.

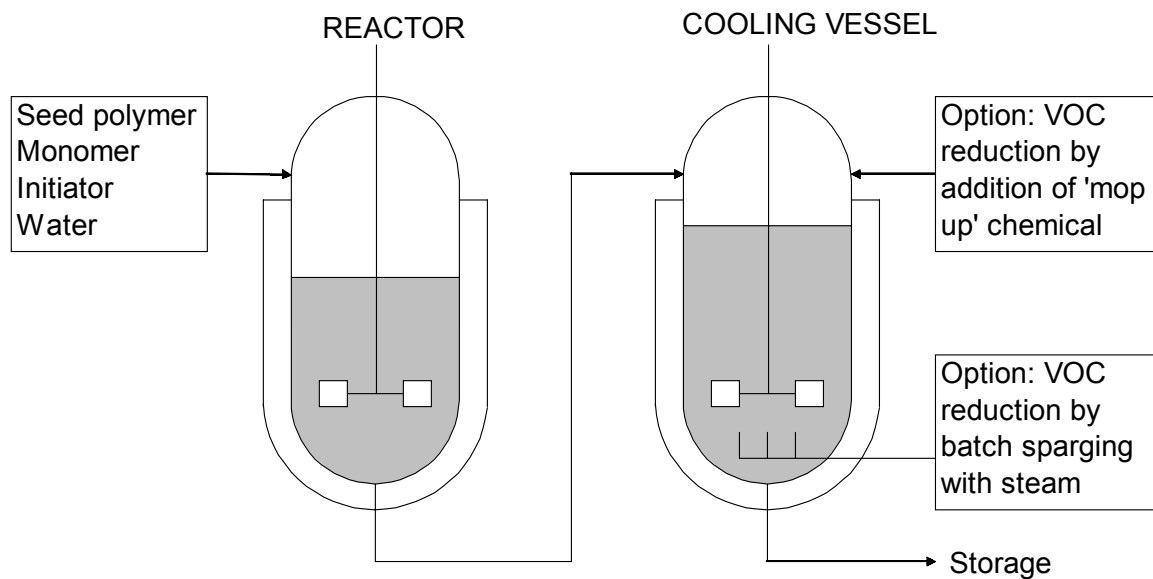
General and specific examples of the basic polymerisation chemistry are outlined in Figure 3. The reactions which produce these aqueous polymers are all instances of Addition Polymerisation of Alkenes.



*Figure 3: Addition Polymerisation of Alkenes*

Figure 4 depicts the typical stages of a batch process for producing aqueous based polymer laticies such as polyvinyl acetate or acrylic polymer.

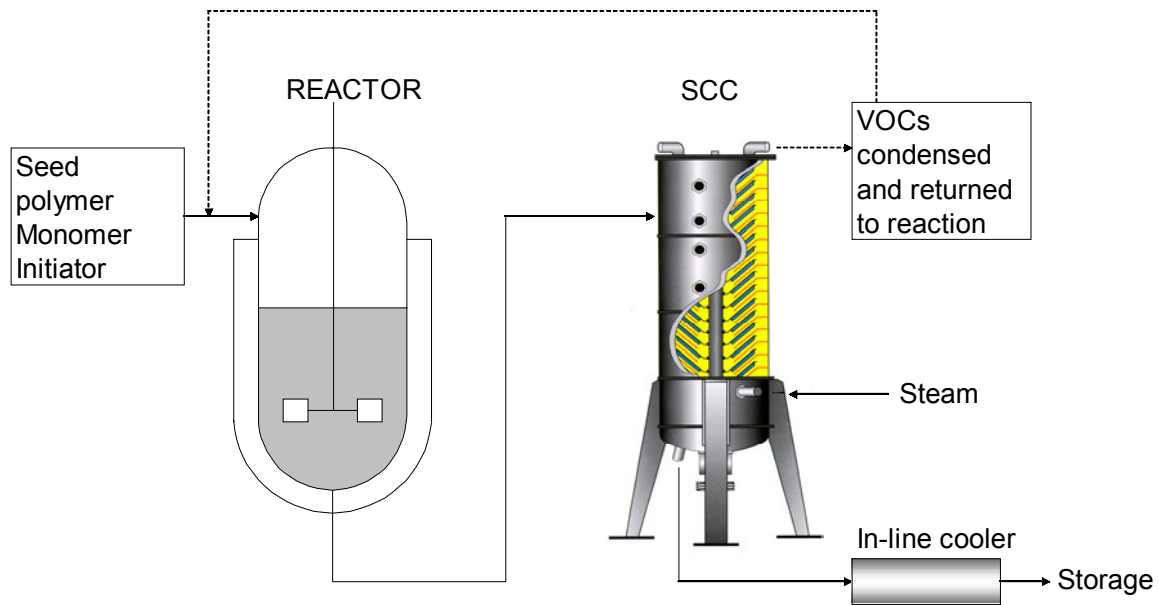




*Figure 4: Typical batch polymer production process*

The main polymerisation reaction is normally preceded by a seed production stage where monomer, water, surfactant and initiator (persulphate catalyst) are mixed and reacted together to produce an initial charge of polymer. More monomer and initiator is then added to complete the batch. Typical temperatures for the reaction are in the range 70 to 80°C with the reaction taking between 3 to 6 hours to complete. At the end of the reaction the batch of polymer must be cooled.

Once the main reaction stage is completed the concentration of residual VOCs needs to be reduced. While in most cases at the end of the main reaction the most abundant VOC will be the key monomer other volatile organic compounds will be present also. The total VOC concentration present at the end of the main polymerisation reaction, prior to a VOC reduction process, will vary according to the specific reaction being completed and the chosen reactor conditions but normally lies in the range 1000 to 10000 ppm.



*Figure 5: Use of SCC for VOC reduction*

Although the polymers used for paint manufacture are often relatively low in viscosity (10 to 100cP) they normally pose difficult handling problems associated with film forming by evaporation, and the equipment used must therefore be capable of being effectively cleaned to remove residues after processing is complete. While the viscosity is described as relatively low it is also true to say that even at 100cP the feed materials give rise to undesirably high pressure drops in conventional continuous stripping columns, and as a result these conventional technologies have not been used commercially. Rather, the solutions considered and ultimately adopted have generally been restricted to the batch steam stripping of the volatiles or batch chemical treatment to 'mop up' the residual volatile compounds. Batch steam stripping crudely involves the sparging of steam directly into the bottom of a vessel containing the material to be stripped of volatiles. While the method and hardware required are relatively simple the process is very inefficient. The SCC offers an efficient, continuous alternative for this essential process step. Integration of the SCC into the process is illustrated in Figure 5.

The comparison between SCC, batch steam stripping and chemical addition for the reduction in VOCs is summarised in Table 1. The SCC can be seen to offer several advantages over both of the alternatives.

Table 1: Comparison of technologies for VOC reduction

Attribute	Batch Steam Stripping	Batch 'mop up' chemical	SCC
Batch or Continuous	Batch	Batch	Continuous
Operating cost	High steam usage	High chemical usage	Low steam usage
Cleanability	Average	Average	Good CIP process
Processing time	High	High	Low

The low steam usage of the SCC, normally representing between 2 and 10% of the total batch volume, is a direct result of the inherently high mass transfer efficiency of the SCC, and can contribute significantly to reducing operating costs. As the only continuous process option the SCC offers potential to further streamline the overall process by development of continuous polymerisation processes incorporating plug flow or cascaded tank reactors. The continuous nature of the SCC combined with the incorporation of in-line cooling leads to the possibility of reducing the processing time by completely removing the necessity for a batch stage after the main polymerisation reaction. If the polymerisation reaction is converted from batch to continuous then significant reductions in work in progress will also be possible.

### High Viscosity

The SCC is particularly well suited to handling viscous materials by virtue of its unique internal design in which the viscous material is spread as a thin film across the rotating cones thus maximising conditions for mass transfer. The effects of viscosity on mass transfer and achievable throughput in the SCC have been discussed by Sykes and Riley [13]. This work puts forward a model approach that supports the empirical observations that both mass transfer and achievable throughput in the SCC are less sensitive to viscosity than in a packed column because of the relative effect of viscosity on available gas flow area.

While many copolymer latices manufactured for paint production have viscosities less than 100 cP, there are many applications for similar copolymers where the viscosity ranges from 1,000cP to 10,000 cP. Such copolymers are used extensively in coatings, specialist paints and adhesives products, and there is a need to reduce the residual volatile organic compound concentration in these high viscosity products, just as in the case of paint. Poly vinyl acetate (PVA) is one example of a copolymer used in the formulation of high viscosity adhesive and coating products.

Figure 6 represents the polymerisation reaction of vinyl acetate to produce polyvinyl acetate (PVA). The main volatile species left over from the polymerisation of PVA is

the monomer vinyl acetate although during the reaction some other residual species are formed, including acetaldehyde, formaldehyde and butanol.

Vinyl acetate

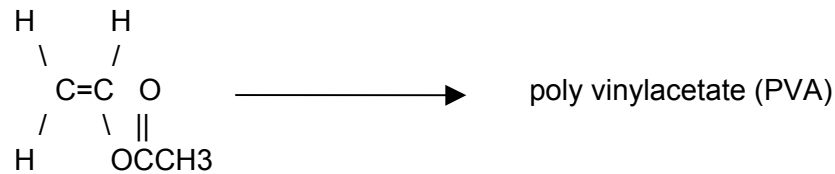


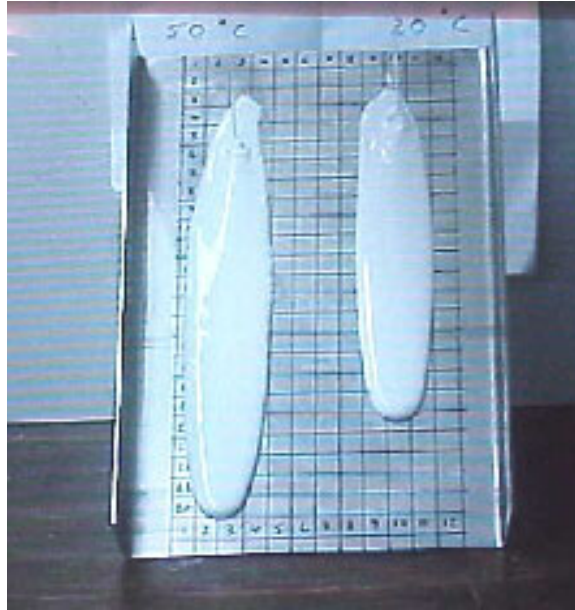
Figure 6: Polymerisation of vinyl acetate

By controlling the specific reactions during the polymerisation PVA copolymer latices can be manufactured to have different film forming properties, viscosity and flexibility when dry. The viscosity range for latices to be used in adhesives and coatings is normally considerably higher than that used for most paint processes and may be in the range 1,000 to 8,000 cP.

Such high viscosity certainly precludes the use of any conventional continuous steam stripping technology and batch steam stripping tends to be the usual approach adopted for the reduction of volatile organic compounds in these materials. The unique internal design of the SCC that has led to successful processing of food products such as banana and mango puree is equally capable of allowing viscous non-food products such as the poly vinyl acetate copolymer described above to be processed.

When any high viscosity material is being considered for processing on the SCC some simple flow tests can be carried out to establish that in principle processing is possible. These tests establish the flow characteristics under gravity down a 45°incline plate. Figure 7 shows a copolymer with a viscosity of approximately 4000 cP flowing down a plate during a simple flow test. In this case the flow test was conducted simultaneously on two samples of the copolymer, one at 20°C the other at 50°C, in order to show the real effect of temperature on viscosity, and thereby on flow characteristics in a low shear situation.

High removal efficiencies have been measured when stripping volatile compounds from highly viscous copolymer latices using the SCC. For example removal rates of greater than 99.6% have been achieved when stripping vinyl acetate from a poly vinyl acetate copolymer with a viscosity of approximately 2000 cP. This removal rate was achieved while using only 25% of the steam normally used by a batch steam stripping process.



*Figure 7: Samples of a copolymer at 50°C and 20°C flowing down a stainless steel plate inclined at 45°. This material has successfully been run through an SCC test rig.*

### **Suspended Solids**

Many slurries containing suspended solids up to 20% are successfully processed in the food sector using SCC technology. This feature is also being put to use in the non-food sector. As well as the polymer applications described above other applications for which it is advantageous to be able to process a slurry are being developed.

For example, the removal or recovery of volatiles from high solids fermentation broth has been successfully demonstrated on the SCC. Often these broths readily form foam and the SCC has been designed to minimise the effect of foam internally with particular attention to de-entrainment features at the top of the column where the feed enters the system.

A more specific example where the SCC can be used to gain advantage in the recovery of volatile compounds from a high solids fermentation broth is the recovery of fermented butanol. Approximately 10% of the world's production of butanol comes from fermentation that typically yields a broth containing between 2 and 2.5% butanol. Conventional distillation columns can be used for the initial separation and concentration of butanol but problems such as foaming, leading to high entrainment, and low separation efficiency are encountered when handling the broth inside these columns. Solvent extraction processes can be used as alternatives to distillation but clearly involve the use of relatively expensive solvents. The SCC can be used to strip the butanol efficiently and directly from the fermentation broth. The ease of cleaning the SCC in place ensures that the column can operate at high efficiency during long continuous production runs.

Another example of where the handling of suspended solids gives rise to new process options is the recovery of volatile compounds from waste water streams. In

the industrial sector the economics of the recovery of solvents from waste water streams containing suspended solids can be improved by use of the SCC. In conventional systems, in which it is necessary to separate the solids prior to solvent recovery, there is an inevitable loss of recoverable solvent with the separated solids. Use of the SCC in this case prevents the loss of solvent and may render the solids separation step unnecessary.

## CONCLUSIONS

The Spinning Cone Column has, over a number of years, evolved into a highly useful and flexible mass transfer device. It is used widely in the food industry in a wide variety of applications.

Features such as cleanability, ability to handle viscous materials and suspended solids, combined with high mass transfer efficiency, have recently led to the development of a number of non-food applications.

It is anticipated that new applications will continue to emerge in the chemical and processing industries, utilising various combinations of the SCC's key operating features. The removal of volatiles from fermentation and pharmaceuticals process streams is anticipated to pose new challenges, and to stimulate further development of SCC technology.

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