

# Spiral Finned Crystallizer for Progressive Freeze Concentration Process

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**ABSTRACT:** Progressive freeze-concentration (PFC) is a method, in which a single ice crystal is formed in the system to remove water from a solution. In this method, the system is much simpler as compared with the conventional method of suspension crystallization, in which many small ice crystals are formed. A small cylindrical test system for PFC was successfully applied for the high quality concentration of fruits flavour solution with high yields of flavour components. Progressive freeze concentration (PFC) has emerged as a viable technology for concentration of liquid solution. In this study, a newly designed spiral finned crystallizer to improve productivity of progressive freeze-concentration process is proposed. The spiral finned crystallizer has high potential to be integrated in a PFC system as an effective system to concentrate a solution and to produce pure ice crystal. This proposed system can also offer an attractive alternative for the food industry as it involves no heating and has high potential in producing highly concentrated solution.

**KEYWORDS:** Progressive freeze concentration, Spiral fins.

## I. INTRODUCTION

Progressive freeze-concentration (PFC) is a method, in which a single ice crystal is formed in the system to remove water from a solution. In this method, the system is much simpler as compared with the conventional method of suspension crystallization, in which many small ice crystals are formed. A small cylindrical test system for PFC was successfully applied for the high quality concentration of fruits flavour solution with high yields of flavour components. No incorporation of flavour components was observed into the ice phase. The concentration yields were compared among the three methods of concentration; evaporation, reverse osmosis, and PFC. PFC showed the highest concentration yield among the three. PFC was also applied for the concentration of apple, tomato, and water melon juices by using tubular ice system for scale-up of PFC. In these cases, partial melting of ice after PFC was effective. By recovering the initial melt fractions with the higher solute concentration, the concentration yield could be improved to a necessary level<sup>[08]</sup>.

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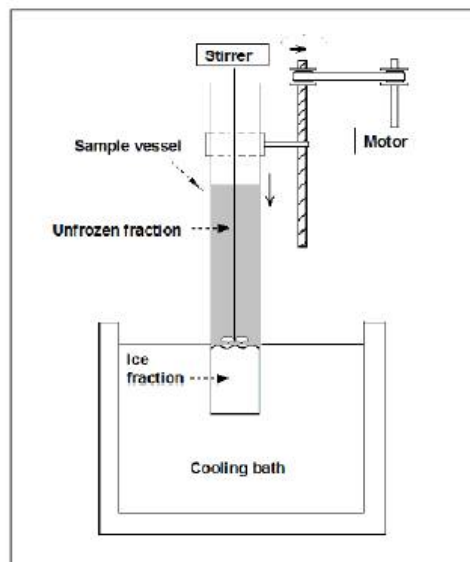


Figure No : 1 test apparatus for progressive freeze-concentration

In the present paper, PFC was applied for the high quality concentration of fruits (pear) flavour solution, apple juice, tomato juice, and water melon juice. When the osmotic pressure of the sample to be concentrated was low, a single step PFC was effective with a high concentration yield for solute components. When the osmotic pressure of the sample was high, some part of the solute was incorporated into the ice phase to reduce the yield. In this case, however, partial melting of ice was effective to improve the concentration yield. Justification of the advantages of FC above has evolved into this research to discover the best FC system design to be employed, which will be focusing on its application for wastewater treatment particularly. In this context, the system should be designed with the attention of producing the purest water possible.[15] There are two available methods for FC, which are suspension freeze concentration (SFC) and progressive freeze concentration (PFC). In SFC, the ice crystals are formed in suspension in the mother liquor, which in this case is the concentrated solution, while in PFC the ice crystal is formed as a single block of ice on the refrigerated surface, as illustrated in Fig.2

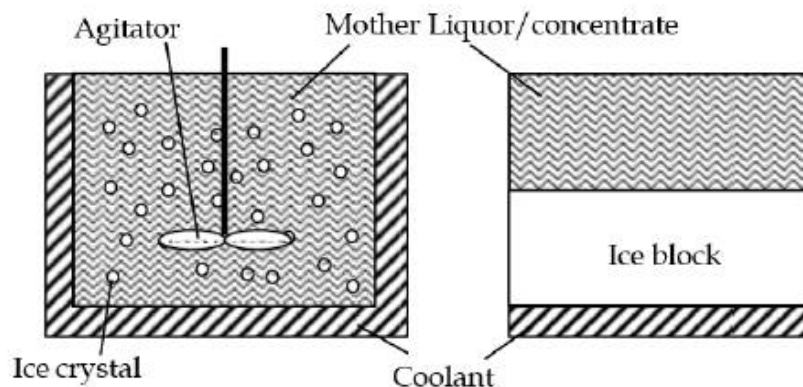


Figure No: 2 (a) Suspension freeze concentration, (b) Progressive freeze concentration

Crystallization is the process of formation of solid crystal precipitating from a solution, or melt. Crystallization is a chemical solid-liquid separation technique, in which mass transfer of a solute from the liquid solution to a pure

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solid crystalline phase occurs. In chemical engineering crystallization occurs in a crystallizer. Crystallization is therefore an aspect of precipitation, obtained through a variation of the solubility conditions of the solute in the solvent, as compared to precipitation due to chemical reaction.

The crystallization process consists of two major events, nucleation and crystal growth. Nucleation is the step where the solute molecules dispersed in the solvent start to gather into clusters, on the nanometre scale (elevating solute concentration in a small region), that becomes stable under the current operating conditions. These stable clusters constitute the nuclei. However, when the clusters are not stable, they dissolve. Therefore, the clusters need to reach critical size in order to become stable nuclei. Such critical size is dictated by the operating conditions (temperature, supersaturation, etc.). It is at the stage of nucleation that the atoms arrange in a defined and periodic manner that defines the crystal structure — note that "crystal structure" is a special term that refers to the relative arrangement of the atoms, not the macroscopic properties of the crystal (size and shape), although those are a result of the internal crystal structure<sup>[1]</sup>

## Five main methods can be used to generate supersaturation:

- 1) Cooling, using the vessel walls, internal coils, or by pumping mother liquor through an external heat exchanger. This is used when solubility changes significantly with temperature and when the feed stream is near saturation at a high temperature.
- 2) Evaporation, by heating the mother liquor or reducing the pressure to form a boiling zone at the top of the vessel. This can be used for a wide range of systems, although it is more energy-intensive than cooling.
- 3) Reaction, where feed streams enter and mix resulting in a chemical reaction generating the product, usually at high levels of supersaturation.
- 4) Drowning out, where a miscible solvent is added resulting in a mixture in which the product is less soluble. This has similar characteristics to reaction crystallization.
- 5) Salting out, where a salt with a common ion is added to precipitate the product from solution. Again, this has similar characteristics to reaction crystallization<sup>[1]</sup>

For crystallization to occur from a solution it must be supersaturated. The solution has to contain more solute entities (molecules or ions) dissolved than it would, contain under the equilibrium (saturated solution).

This can be achieved by various methods, with

- (1) Solution cooling,
- (2) Addition of a second solvent to reduce the solubility of the solute (technique known as anti-solvent)
- (3) Chemical reaction and
- (4) Change in pH being the most common methods used in industrial practice.

Other methods, such as solvent evaporation, can also be used.

## II. RELATED WORK (DESIGN)

**Inner tube:** Feed is flows through inner tube made of stainless steel. The measure function of inner tube is to transfer heat from inner side to outer side. Inlet is provided at top of tube and outlet for concentrated juice is at bottom.



Figure No: 3 Specifications of Inner Tube

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Inner diameter (d): 35mm

Outer diameter (D): 37mm

Height (h): 480mm

Thickness of wall: 1mm

Calculation of surface area

Total surface area can be calculated by,

$$A = 2\pi rh + 2\pi r^2$$

$$= (2 \times 3.14 \times 18.5 \times 480) + (2 \times 3.14 \times 18.5^2)$$

$$= 57945.73 \text{ mm}^2 \text{ or } 579.4573 \text{ cm}^2$$

**Fin:** Fins are surfaces that extend from an object to increase the rate of heat transfer to or from the environment by increasing convection. The amount of conduction, convection, or radiation of an object determines the amount of heat it transfers. A square long fin is used and is welded spirally to the inner tube at outer surface. Active surface area is calculated for three edges only.

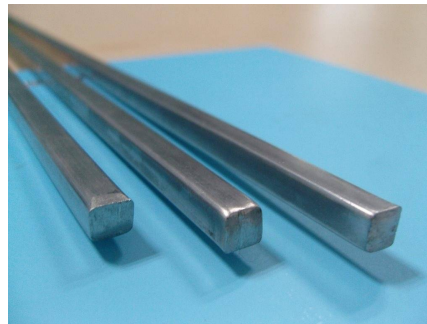


Figure No: 4 Specifications of Fin

Specifications:

Width (w): 10 mm

Thickness (t): 10 mm

Length (l): 410 mm

No of Fins: 6

Calculation of surface area

1) Total surface area

$$A = 2wl + 2tl + 2wl$$

$$= 2(10 \times 10) + 2(10 \times 410) + 2(10 \times 410)$$

$$= 99600 \text{ mm}^2 \text{ or } 996 \text{ cm}^2$$

2) Active surface area

$$A = \frac{3}{4} (\text{Total surface area})$$

$$= 747 \text{ cm}^2$$

**Jacket (outer tube) :** Jacket is provided for the coolant which made up of stainless steel. Two valve are joined at Top and bottom. Coolant is filled in annular portion between inner tube and outer tube.

**Specifications:** Inner diameter: 97 mm, Outer diameter: 100 mm, Height: 430 mm

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Figure No: 5 Specifications of Jacket (outer tube)

**-Total Active Heat Transfer Area:**

$$AT = \text{Active area of Inner tube} + \text{Active area of fin} = 576.9673 + 74 = 1323.9673 \text{ cm}^2$$

**-Model specification:** Inner tube capacity = 450 ml ,Outer jacket capacity = 2500 ml

### III .EXPERIMENTAL PROCEDURE

**Feed:** Feed is consisting of sugarcane juice containing higher amount of water. Chemical composition of sugarcane juice differences between varieties and locations can be established. Sugarcane juice consists of a sucrose solution containing soluble and insoluble impurities (i.e. On sucrose species). It's was also slightly acid with the pH between 4.8 to 5.2, and sucrose 18.45 % w/w. the ability of sucrose to crystallize is depends upon sugar content and pH.



Figure No: 6 Fresh feed of sugarcane

**Coolant:** Brine solution is used as coolant for processing freezing of sugarcane juice. Brine solution is nothing but the mixture of ice and salt. Adding salt to the ice/water mix causes temperature drop that slows the melting rate and increases the freezing rate. The net result is that the ice melts more and more slowly after the initial addition of salt.

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Figure No: 7 Brine solutions

NaCl, wt.%	Freezing point (°C)	Density (g/cm <sup>3</sup> )
0	0	0.99984
1	-0.59	1.0053
2	-1.19	1.0125
3	-1.79	1.0196
4	-2.41	1.0268
5	-3.05	1.034
6	-3.7	1.0413
7	-4.38	1.0486
8	-5.08	1.0559
9	-5.81	1.0633
10	-6.56	1.0707
12	-8.18	1.0857
14	-9.94	1.1008
16	-11.89	1.1162
18	-14.04	1.1319
20	-16.46	1.1478

Table no: 1 Relation between freezing point of brine solution & NaCl wt%

- General procedure:**
1. Mount the reactor on stand.
  2. Make sure all the valves are closed.
  3. Fill the brine solution in outer jacket.
  4. Check the temperature of brine solution.
  5. Check the temperature of feed.
  6. Fed the feed (sugarcane juice) to the reactor.
  7. Note down the temperature of feed at particular time interval.
  8. Perform batch operation for different time span.
  9. After the individual run remove the juice from reactor. Analyse the water content Removed i.e. ice crystals on inner wall of tube and remained water in juice.
  10. Generate data of result.

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Figure No: 8 Experimental Setup

## IV. EXPERIMENTAL RESULT

**Batch operation I (for 5 min) :** Initially 10% brine solution made from 2 kg Ice and 200 grams of NaCl. Which cause reduction in temperature of brine solution by 6°C. feed is introduced in inner tube. The quantity of feed taken is 400 ml. The resulting setup is kept for batch crystallisation for 5 min. after 5min the product is removed from bottom valve. The quantity of bottom product is measured. The crystals formed during the batch process are removed. For First run the feed is taken at 18°C and run is performed for 5 min in batch process. Next Runs are carried out at 10°C, 5°C, and 2°C feed temperature respectively. And fresh coolant is refilled for every run with same composition. As shown in following figure 8, water crystals are removed formed during freezing of sugarcane juice. These crystals are formed at inner wall of tube when temperature of feed material reaches to its freezing point i.e. water's freezing point.



Figure No : 9 Water Crystal Formed

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Sr. No	Time (Min)	Feed Temp T (°C)	Coolant Temp Tc (°C)	Feed quantity (ml)	Bottom product (ml)	Water removed (ml) (Crystals)	Ice production g/m <sup>2</sup> s
01	5	18	-6	400	377	23	0.579068
02	5	14	-6	400	373	27	0.679775
03	5	12	-6	400	367	33	0.830836
04	5	10	-6	400	357	43	1.082605
05	5	8	-6	400	352	48	1.208489
06	5	5	-6	400	341	59	1.485434
07	5	2	-6	400	323	68	1.712026

Table no: 2Result of First Batch operation ( 5 minutes)

**Batch operation II (for 10 min) :** Initially 10% brine solution made from 2 kg Ice and 200 grams of NaCl. Which causes reduction in temperature of brine solution by 6°C. feed is introduced in inner tube. The quantity of feed taken is 400 ml. The resulting setup is kept for batch crystallisation for 10 min. After 10 min the product is removed from bottom valve. The quantity of bottom product is measured. The crystals formed during the batch process are removed. For First run the feed is taken at 18°C and run is performed for 10 min in batch process. Next Runs are carried out at 10°C, 5°C, and 2°C feed temperature respectively. And fresh coolant is refilled for every run with same composition.

Sr. No	Time (Min)	Feed Temp T (°C)	Coolant Temp Tc (°C)	Feed quantity (ml)	Bottom product (ml)	Water removed (ml) (Crystals)	Ice production g/m <sup>2</sup> s
01	10	18	-6	400	365	35	0.440595
02	10	14	-6	400	355	45	0.566479
03	10	12	-6	400	348	52	0.654598
04	10	10	-6	400	337	63	0.793071
05	10	8	-6	400	330	70	0.88119
06	10	5	-6	400	319	81	1.019663
07	10	2	-6	400	308	92	1.158135

Table no: 3Result of First Batch operation ( 10 minutes)

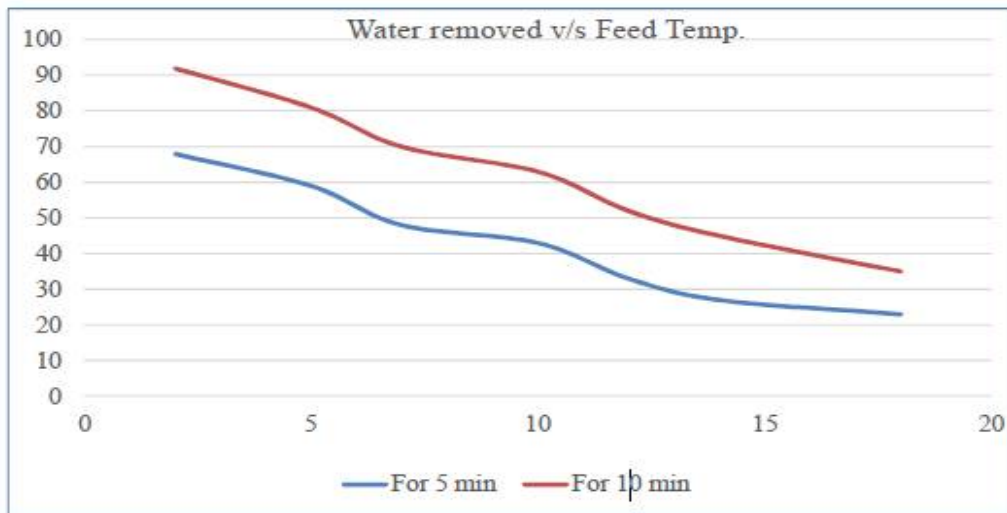


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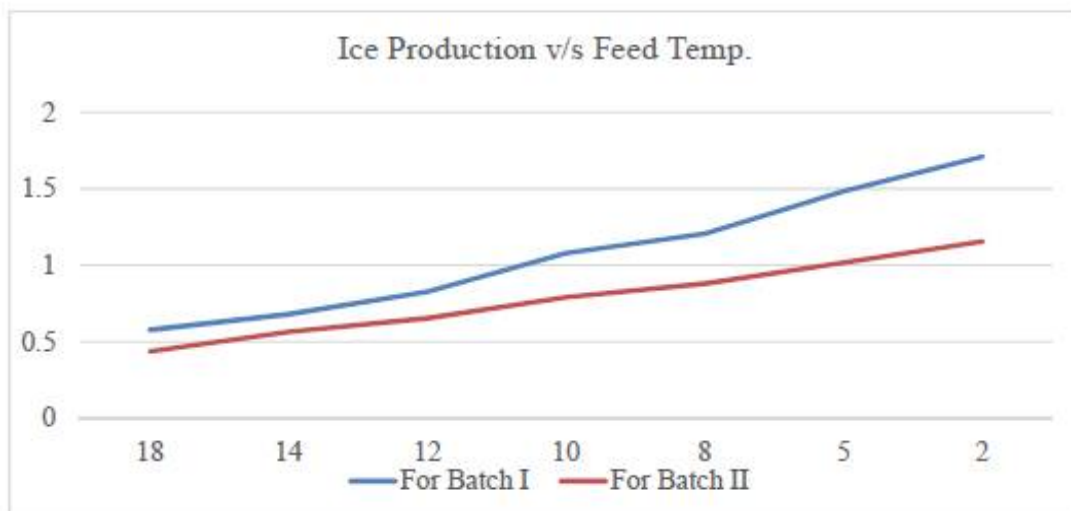
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**Graphical Representation of water removed with respect to feed Temperature:** Freezing of juice is carried out in batch operation keeping coolant temperature constant and varying feed temperature which causes the amount of water crystallisation.



Graph No: 1 Relationship between ice production in crystalliser and temperature of feed with water removed



Graph No: 2 Relationship between ice production in crystalliser and temperature of feed

## V .CONCLUSION

This study demonstrated the feasibility of the idea and design for the spiral finned crystallizer. The effectiveness of spiral finned freeze concentrator system was experimentally confirmed through the concentration of sugarcane juice. The operating conditions that influence the concentration of sugarcane juice were tested using the spiral finned crystallizer. The water crystals formed were found to be dependent on the Coolant Temperature, Feed Temperature and

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Residence Time. It is important to operate the system with precision in order to form pure ice and to recover the solute. The lowest value of water removed from juice is found to be 23mL and the highest value of water removed is 92 mL per 400 mL of feed. The maximum ice production attained was 1.15 g/m<sup>2</sup>s in accordance with coolant temperature. Coolant temperature of -6 °C, residence time 10 min and keeping feed temperature at minimum results in higher amount of crystallisation. By the study we come to know total Heat Transfer area plays measure role in concentrating of juice. In over-all, the results show the feasibility of the s system and provide excellent idea of the operating conditions that can be used to concentrate any fruit juice. The PFC system is a potential technique to produce high quality concentrated solution, with the advantage of the simplicity of the equipment.

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