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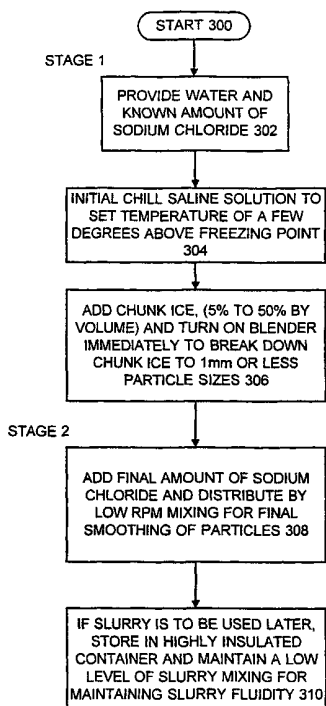
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(54) Title: METHODS AND APPARATUS FOR PRODUCING PHASE CHANGE ICE PARTICULATE SALINE SLURRIES



(57) Abstract: A phase change particulate saline slurry and methods and apparatus are provided for producing phase change particulates saline slurries. In one method for producing phase change particulates saline slurries (300), water and a first set amount of sodium chloride are provided to produce a saline solution (302). The saline solution is cooled to a set temperature (304). A selected percentage of chunk ice is broken into ice particles having a small size (306). Next a second set amount of sodium chloride is added and distributed for smoothing of the ice particles (308). If the slurry is to be used later, the slurry is stored in a highly insulated container while maintaining low level of slurry mixing for maintaining optimum slurry fluidity as indicated in block (310). The total saline solution concentrations resulting from the total of the first set amount and the second set amount of added sodium chloride are preferably in the range of about 0.5 % to 6.0 %. The loadings or percentage of ice particles are preferably in the range of 5 % to 50 % and have a size of about 1 mm or less and a generally smooth shape.



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## **METHODS AND APPARATUS FOR PRODUCING PHASE CHANGE ICE PARTICULATE SALINE SLURRIES**

This application claims the benefit of prior filed copending provisional application filed August 2, 1999, by Kenneth E. Kasza et al., provisional  
5 application number 60/146,753 and entitled METHOD FOR INDUCING HYPOTHERMIA. The subject matter of the above-identified copending provisional application is incorporated herein by reference.

### **CONTRACTUAL ORIGIN OF THE INVENTION**

The United States Government has rights in this invention pursuant to  
10 Contract No. W-31-109-ENG-38 between the United States Government and Argonne National Laboratory.

### **Field of the Invention**

The present invention relates to methods and apparatus for producing  
15 phase change ice particulate saline slurries of very high fluidity, cooling capacity and stability.

### **Description of the Related Art**

Phase change slurries in the form of high concentrations of small ice  
20 particles in a liquid carrier have dramatically increased coolant capacity as compared to other liquids such as single phase water or other liquids involving no heat of fusion effects. Phase change ice slurries developed by

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the present inventor Kenneth E. Kasza have been used for cooling in large building complexes. The development of ice slurries by the inventor for cooling buildings has shown that ice particles suspended in water or other carrier liquid, if engineered to have the correct characteristics, can be pumped as readily as water and are stable for significant periods of time without agglomeration where ice particles freeze together or entangle in clusters. The cooling capacity of such a slurry can be 5 to 10 times, depending on the particular loading in the carrier liquid, that of an equal amount of water which exhibits only sensible heat cooling capacity. For use in cooling buildings, the particles preferably are small relative to the conduit diameter, not loaded to a level of more than 30% ice in order to enhance delivery to the target cooling zone, and relatively smooth to avoid particle entanglement and formation of large clusters. Small additions of certain types of chemicals, such as freezing point depressants, when added to a slurry during an appropriate time when making the slurry, have been shown to dramatically improve the fluidity and storability of the slurry by altering the microscale features (smoothing) of the individual particles comprising the slurry.

A need exists for an improved method and apparatus for producing phase change particulate saline slurries. It is desirable to produce phase change particulate saline slurries, with high cooling capacity, fluidity, and storability, for example for use to induce targeted protective hypothermia of human organs/tissue during medical treatment.

It is an object of the present invention to provide methods and apparatus for producing phase change particulate saline slurries.

It is an object of the present invention to provide an improved phase change particulate saline slurry.

### **Summary of the Invention**

In brief, a phase change particulate saline slurry and methods and apparatus are provided for producing phase change particulate saline

slurries. One method for producing phase change particulate saline slurries includes the steps of providing a liquid with a set percentage freezing point depressant to form a first solution, such as, a set percentage saline solution; cooling the first solution to a set temperature to produce ice particles; and  
5 increasing an ice particle concentration under controlled temperature for a period of time to provide a set ice particle concentration for the phase change particulate saline slurry.

In another method for producing phase change particulate saline slurries, water and a first set amount of sodium chloride are provided to  
10 produce a saline solution. The saline solution is cooled to a set temperature. A selected percentage of chunk ice is added to the saline solution and the chunk ice is broken into ice particles. The ice particles have a small size. Next a second set amount of sodium chloride is added and distributed for smoothing of the ice particles.

15 A phase change particulate saline slurry includes a water and sodium chloride solution. The sodium chloride is provided in a range between about 0.5% to 6.0%. A percentage of ice particles is provided in the range between about 5% to 50%. The ice particles have a size of about 1 mm or less than 1 mm; and the ice particles have a generally smooth shape.

20 In accordance with features of the invention, the total saline solution concentrations resulting from the total of the first set amount and the second set amount of added sodium chloride are preferably in the range of about 0.5% to 6.0%. The loadings or percentage of ice particles are preferably in the range of 5% to 50%. The resulting slurry if to be stored for later use is  
25 stored in a highly insulated container to reduce melt out of ice particles and temperature gradient in the stored slurry which can cause ice particle freezing together and reduce fluidity.

### **Brief Description of the Drawings**

30 The present invention together with the above and other objects and advantages may best be understood from the following detailed description

of the preferred embodiments of the invention illustrated in the drawings, wherein:

FIG. 1 is a block diagram representation illustrating exemplary apparatus for producing phase change particulate saline slurries;

5           FIG. 2 is a flow chart illustrating exemplary steps for producing phase change particulate saline slurries; and

FIG. 3 is a flow chart illustrating exemplary steps for producing phase change particulate saline slurries in accordance with a preferred embodiment of the invention.

## 10       **Detailed Description of the Preferred Embodiments**

Having reference now to the drawings, in FIG. 1 there is shown apparatus for producing phase change particulate saline slurries in accordance with an embodiment of the invention and generally designated by reference character 100. A container 102 contains a selected percentage saline solution 104. A chiller device 106 is provided for cooling the saline solution 104. A mixer 110 and an associated rotator 112, such as a variable speed mixer or blender, is provided for processing the phase change particulate saline slurries in accordance with methods illustrated and described with FIGS. 2 and 3. The method illustrated and described with respect to FIG. 3 is the preferred embodiment of the invention.

In accordance with features of the invention, saline solution concentrations are preferably in the range of about 0.5% to 6.0% and the loadings, or percentage of ice particles, are preferably in the range of 5% to 50%. The phase change particulate saline slurry of the preferred embodiment is stable or storable, fluid and highly loaded ice particle medical grade slurry. A phase change particulate saline slurry of the preferred embodiment advantageously can be used to induce targeted protective hypothermia during medical treatment. The medical use of slurries for cooling in general requires slurries of higher fluidity than those currently used

in building cooling due to the smaller diameter of slurry delivery tubing, needles and organ/blood vessel flow passages.

In accordance with features of the invention, both chemical and thermal alteration of ice particles can be used together or individually. By introducing a small amount of thermally induced ice particle melting, such as by adding a warmer fluid at an appropriate time during slurry particle formation, produces beneficial particle smoothing. The improved slurries of the invention are based upon developments of ice slurries by the inventor at Argonne National Laboratory.

Referring to FIG. 2, there are shown exemplary steps for producing phase change particulate saline slurries in accordance with a first embodiment of the invention starting at a block 200. First a set percentage saline solution or other freezing point depressant is provided to form a first solution as indicated in a block 202. For example, the saline solution is a sodium chloride aqueous solution serving as a freezing point depressant. The saline solution alters the nature of the ice crystals formed during the subcooling stage of the solution and subsequent flash freezing which causes homogenous ice particle nucleation/formation in the bulk solution when the solution is subcooled to below its freezing point. Using, for example, a 0.9% saline solution, a slurry can be formed both in saline solution confined in a container or directly in a plastic medical IV injection bag. The saline solution is cooled to a point where ice crystals form as indicated in a block 204. For the 0.9% saline solution, the freezing point for this concentration of sodium chloride is  $-0.5^{\circ}\text{C}$ . In contrast to pure water, the saline solution in the beaker and the medical injection bag forms very small separated ice crystals of size less than 0.1 mm initially. These very small ice crystals grow to approximately 0.2 mm or 0.3 mm as the ice crystal loadings grows with time after initial nucleation of the solution under a low level of agitation or mixing. The slurry is allowed to increase in ice crystal concentration for a set period of time while providing low level mixing to lessen dendrite particle growth, for example, about 15 minutes, yielding a selected ice crystal concentration of approximately 5% to 50% as indicated in a block 206. In the produced slurry, the ice particles are generally smaller and less dendritic than if formed

in pure water due to the presence of the sodium chloride. This slurry readily flows through 3 mm diameter tubing.

Referring to FIG. 3, there are shown exemplary steps for producing phase change particulate saline slurries in accordance with another preferred embodiment of the invention starting at a block 300. In a first stage, water and a known amount of sodium chloride or another type of freezing point depressant or combinations thereof, are provided to provide a desired saline solution as indicated in a block 302.

For example, the amount of sodium chloride may be in a range between about 0.5% weight of sodium chloride to 5% weight of sodium chloride in water. The more sodium chloride used provides a lower freezing point of the solution. At 0.5% by weight of sodium chloride in water, the equilibrium temperature is approximately  $-0.3^{\circ}\text{C}$ . At 5% by weight of sodium chloride in water, the equilibrium temperature is approximately  $-4^{\circ}\text{C}$  to  $-5^{\circ}\text{C}$ . The more sodium chloride used provides smoother ice particles up to a point of diminishing returns which depending on particle initial roughness is in the range of 4%-7%.

Next the saline solution produced at block 302 is chilled to a set temperature, such as several degrees above the freezing point of the saline solution for example, approximately  $7^{\circ}\text{C}$  to  $10^{\circ}\text{C}$  as indicated in a block 304. This cooling step facilitates mixing and thermal induced smoothing of small particles formed during ice chunk break down to the particle size needed for the application. Then chunk ice is added, for example 50% by volume and a high speed blender is turned on immediately to break down the chunk ice to particles of a size of 1 mm or less as indicated in a block 306. The very small ice particles have a generally smooth, globular or spherical shape with some remaining particle roughness.

In a second stage as indicated in a block 308, a final amount of sodium chloride is added and the blender is turned on for low speed mixing to prevent additional particle break down and to distribute the added sodium chloride into the slurry for final smoothing of the small ice particles. If the

slurry is to be used later, the slurry is stored in a highly insulated container while maintaining low level of slurry mixing for maintaining optimum slurry fluidity as indicated in a block 310.

In accordance with features of the invention, the two stage process of  
5 FIG. 3 produces an improved phase change particulate saline slurry that readily flows through a 14 GA needle. This improved phase change particulate saline slurry readily flows through plastic tubing as small as 1 mm in diameter. The total saline solution concentrations resulting from the total amount of added sodium chloride at blocks 302 and 308 are preferably in  
10 the range of about 0.5% to 6.0%. The loadings, or percentage of ice crystals, are preferably in the range of 5% to 50%. The first method of FIG. 2 produces more elongated and dendritic, rough particles which render the slurry of lower fluidity, but depending on use can be adequate. It should be understood that the present invention is not limited to the use of a sodium  
15 chloride saline solution. The methods of the invention are applicable to slurries involving water and other types of freezing depressants, various saline solutions with other types of salts or combinations. For medical treatment, the chemicals must be compatible with human organs/tissue.

While the present invention has been described with reference to the  
20 details of the embodiments of the invention shown in the drawing, these details are not intended to limit the scope of the invention as claimed in the appended claims.



**Claims**

What is claimed is:

- 1           1.     A method for producing phase change particulate saline  
2 slurries comprising the steps of:  
3           providing a liquid with a set percentage freezing point depressant to  
4 form a first solution;  
5           cooling said saline solution to a set temperature to produce ice  
6 particles; and  
7           increasing an ice particle concentration under controlled temperature  
8 for a period of time to provide a set ice particle concentration for the phase  
9 change particulate saline slurry.
  
- 1           2.     A method for producing phase change particulate saline  
2 slurries as recited in claim 1 wherein the step of providing liquid with a set  
3 percentage freezing point depressant to form a first solution includes the  
4 step of providing water with a set percentage saline solution in a range  
5 between about 0.5% to 6.0%.
  
- 1           3.     A method for producing phase change particulate saline  
2 slurries as recited in claim 1 wherein the step of increasing an ice particle  
3 concentration to provide a set ice particle concentration for the phase  
4 change particulate saline slurry includes the step of providing percentage of  
5 ice particles in the range of about 5% to 50%.

1           4.     A method for producing phase change particulate saline  
2 slurries as recited in claim 1 wherein the step of providing cooling said saline  
3 solution to a set temperature to produce ice particles includes the step of  
4 subcooling said saline solution below a freezing point and wherein the step  
5 of increasing an ice particle concentration under controlled temperature for a  
6 period of time to provide a set ice particle concentration for the phase  
7 change particulate saline slurry includes the step of increasing said ice  
8 particle concentration under said controlled temperature below said freezing  
9 point for a period of time to provide said set ice particle concentration for the  
10 phase change particulate saline slurry.

1           5.     A method for producing phase change particulate saline  
2 slurries as recited in claim 1 wherein the step of providing a set percentage  
3 saline solution includes the step of providing said set percentage of sodium  
4 chloride saline solution in a range between about 0.5% to 6.0%.

1           6.     A method for producing phase change particulate saline  
2 slurries comprising the steps of:  
3           providing water and a first set amount of sodium chloride to produce a  
4 saline solution;  
5           cooling said saline solution to a set temperature;  
6           adding a selected percentage of chunk ice to said saline solution and  
7 breaking said chunk ice into ice particles; said ice particles having a small  
8 size; and  
9           adding a second set amount of sodium chloride and distributing said  
10 added second set amount of sodium chloride for smoothing of said ice  
11 particles.

1           7.     A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said steps of providing water and a first  
3 set amount of sodium chloride to produce a saline solution and adding a  
4 second set amount of sodium chloride provide a saline solution in a range  
5 between about 0.5% to 6.0%.

1           8.     A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of adding a selected  
3 percentage of chunk ice to said saline solution provides a percentage of ice  
4 particles in the range of about 5% to 50%.

1           9.     A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of breaking said chunk ice  
3 into ice particles includes the step of utilizing a high speed blender and  
4 breaking said chunk ice into ice particles.

1           10.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said ice particles have a size of about 1  
3 mm or less than 1 mm.

1           11.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of cooling said saline solution  
3 to a set temperature includes the step of cooling said saline solution to a  
4 temperature above a freezing point of about 7°C to 10°C to provide thermal  
5 smoothing of particles during break down of said chunk ice.

1           12.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said first set amount of sodium chloride  
3 and said second set amount of sodium chloride are approximately equal and  
4 together provide a saline solution in a range between about 0.5% to 6.0%.

1           13.    A phase change particulate saline slurry comprising:  
2           a water and sodium chloride solution; said sodium chloride provided  
3 in a range between about 0.5% to 6.0%; and  
4           a percentage of ice particles in the range between about 5% to 50%;  
5 said ice particles having a size of about 1 mm or less than 1 mm; and said  
6 ice particles having a generally smooth shape.

1           14. A phase change particulate saline slurry as recited in claim 13  
2 is formed by the steps of:  
3           providing water and a first set amount of sodium chloride to produce a  
4 saline solution;  
5           cooling said saline solution to a set temperature;  
6           adding a selected percentage of chunk ice to said saline solution and  
7 breaking said chunk ice into ice particles; said ice particles having a small  
8 size; and  
9           adding a second set amount of sodium chloride and distributing said  
10 added second set amount of sodium chloride for smoothing of said ice  
11 particles.

1           15. A phase change particulate saline slurry as recited in claim 13  
2 is formed by the steps of:  
3           providing a set percentage saline solution;  
4           cooling said saline solution to a set temperature to produce ice  
5 particles; and  
6           increasing an ice particle concentration under controlled temperature  
7 for a period of time to provide a selected particle concentration for the phase  
8 change particulate saline slurry.

1           16. Apparatus for producing phase change particulate saline  
2 slurries comprising:  
3           a container for containing a saline solution including water and a first  
4 set amount of sodium chloride;  
5           a cooler for cooling said saline solution to a set temperature;  
6           means for adding a selected percentage of chunk ice to said saline  
7 solution and means for breaking said chunk ice into ice particles; said ice  
8 particles having a small size; and  
9           means for adding a second set amount of sodium chloride and means  
10 for distributing said added second set amount of sodium chloride for  
11 smoothing of said ice particles.

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**AMENDED CLAIMS**

[received by the International Bureau on 29 December 2000 (29.12.00);  
original claim 13 cancelled; original claims 14 and 15 amended and renumbered as claims  
13-14 ; claim 16 renumbered as claim 15  
other claims unchanged (3 pages)]

1           8.     A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of adding a selected  
3 percentage of chunk ice to said saline solution provides a percentage of ice  
4 particles in the range of about 5% to 50%.

1           9.     A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of breaking said chunk ice  
3 into ice particles includes the step of utilizing a high speed blender and  
4 breaking said chunk ice into ice particles.

1           10.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said ice particles have a size of about 1  
3 mm or less than 1 mm.

1           11.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said step of cooling said saline solution  
3 to a set temperature includes the step of cooling said saline solution to a  
4 temperature above a freezing point of about 7°C to 10°C to provide thermal  
5 smoothing of particles during break down of said chunk ice.

1           12.    A method for producing phase change particulate saline  
2 slurries as recited in claim 6 wherein said first set amount of sodium chloride  
3 and said second set amount of sodium chloride are approximately equal and  
4 together provide a saline solution in a range between about 0.5% to 6.0%.

1           13. A phase change particulate saline slurry comprising:  
2           a water and sodium chloride solution; said sodium chloride provided  
3           in a range between about 0.5% to 6.0%; and  
4           a percentage of ice particles in the range between about 5% to 50%;  
5           said ice particles having a size of about 1 mm or less than 1 mm; and said  
6           ice particles having a generally smooth shape; and said phase change  
7           particulate saline slurry formed by the steps of:  
8           providing water and a first set amount of sodium chloride to produce a  
9           saline solution;  
10          cooling said saline solution to a set temperature;  
11          adding a selected percentage of chunk ice to said saline solution and  
12          breaking said chunk ice into ice particles; said ice particles having a small  
13          size; and  
14          adding a second set amount of sodium chloride and distributing said  
15          added second set amount of sodium chloride for smoothing of said ice  
16          particles.

1           14. A phase change particulate saline slurry comprising:  
2           a water and sodium chloride solution; said sodium chloride provided  
3           in a range between about 0.5% to 6.0%; and  
4           a percentage of ice particles in the range between about 5% to 50%;  
5           said ice particles having a size of about 1 mm or less than 1 mm; and said  
6           ice particles having a generally smooth shape; and said phase change  
7           particulate saline slurry formed by the steps of:  
8           providing a set percentage saline solution;  
9           cooling said saline solution to a set temperature to produce ice  
10          particles; and  
11          increasing an ice particle concentration under controlled temperature  
12          for a period of time to provide a selected particle concentration for the phase  
13          change particulate saline slurry.

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1           15.    Apparatus for producing phase change particulate saline  
2 slurries comprising:  
3           a container for containing a saline solution including water and a first  
4 set amount of sodium chloride;  
5           a cooler for cooling said saline solution to a set temperature;  
6           means for adding a selected percentage of chunk ice to said saline  
7 solution and means for breaking said chunk ice into ice particles; said ice  
8 particles having a small size; and  
9           means for adding a second set amount of sodium chloride and means  
10 for distributing said added second set amount of sodium chloride for  
11 smoothing of said ice particles.

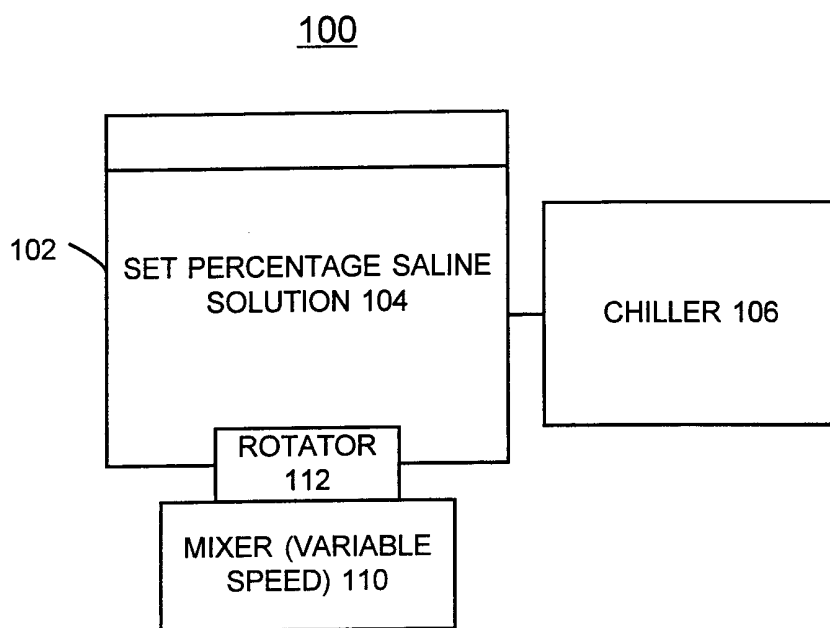


FIG. 1



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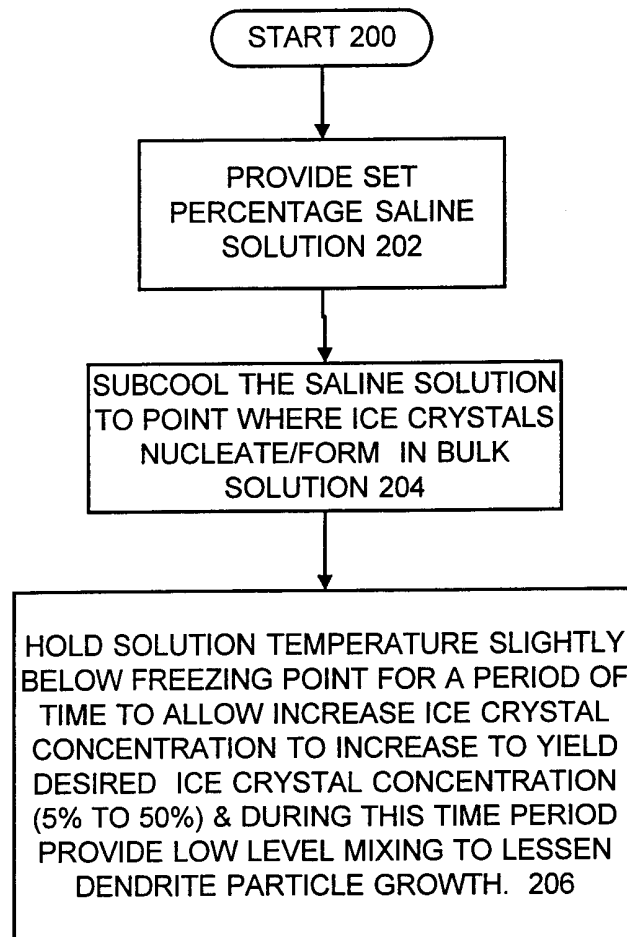


FIG. 2

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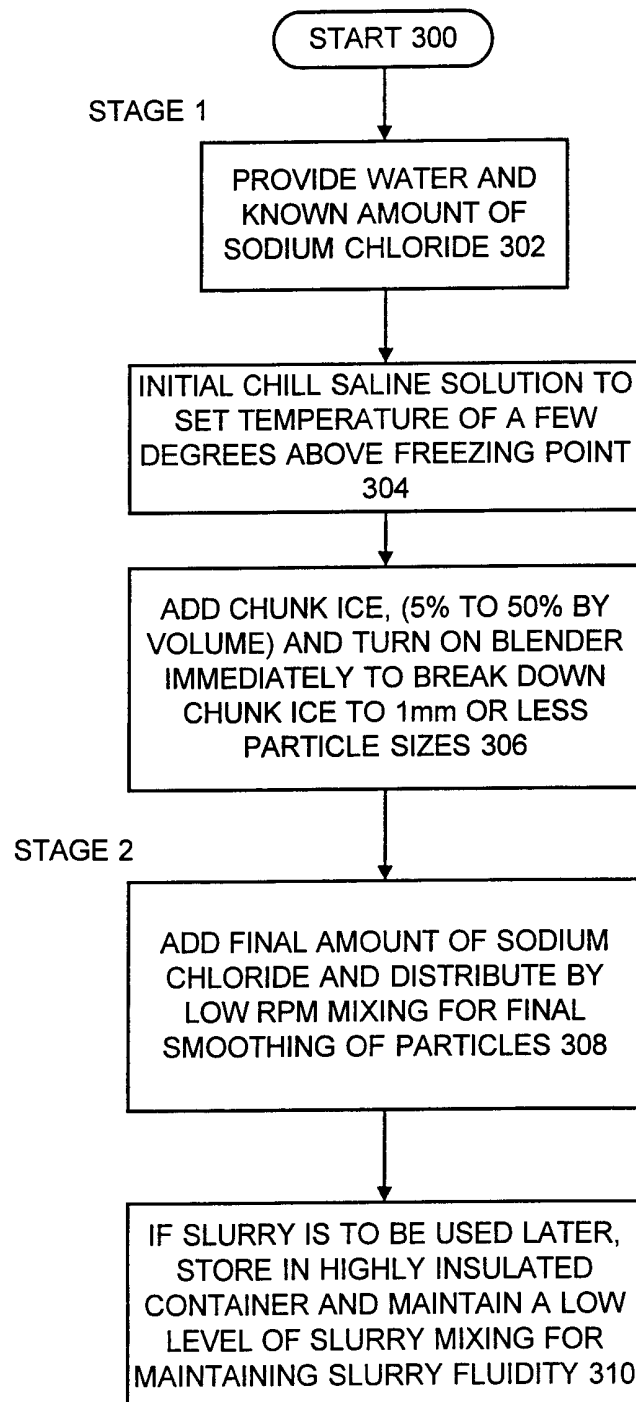


FIG. 3

INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US00/21000

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(7) :F25C 1/00; C09K 5/02  
US CL :106/13; 252/70; 62/66, 68, 75, and 340  
According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)  
U.S. : 106/13; 252/70; 62/66, 68, 75, and 340

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3,987,211 A (DUNN et al) 19 OCTOBER 1976, abstract, col. 2, lines 41-63, and the examples.	13
A	US 4,750,336 A (MARGEN) 14 JUNE 1988.	1-16
A	US 4,914,921 A (KNODEL) 10 APRIL 1990.	1-16
A	US 4,596,120 A (KNODEL et al) 24 JUNE 1986.	1-16
A, P	JP 11-335660 A (HITACHI ZOSEN CORP.) 07 DECEMBER 1999.	1-15

Further documents are listed in the continuation of Box C.  See patent family annex.

* Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search 17 OCTOBER 2000	Date of mailing of the international search report 21 NOV 2000
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