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(71) Applicant (for all designated States except US):
BIOAMBER, S.A.S. [FR/FR]; Route de Bazancourt,
F-51110 Pomacle (FR).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **DUNUWILA, Dilum** [US/US]; 53 Dorann Av., Princeton, NJ 08540 (US). **BERNIER, Roger, Laurent** [CA/CA]; 7-730 Lusignan, Montréal, Québec H3C 1Y9 (CA).

(74) Agent: **ROBIC**; Centre CDP Capital, 1001, Square-Victoria, Bloc E, 8th Floor, Montréal, Québec H2Z 2B7 (CA).

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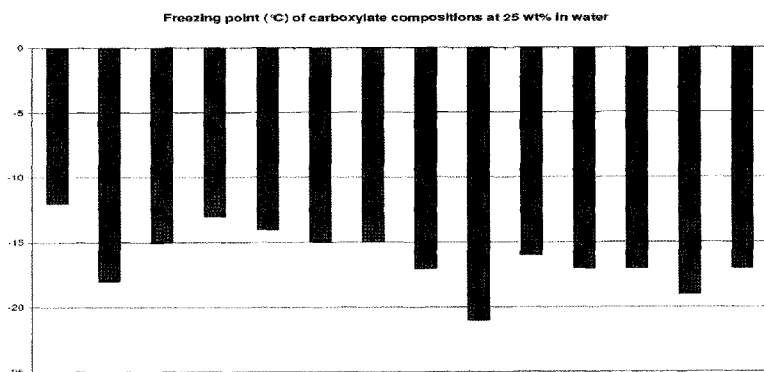


Fig. 7

(57) Abstract: A composition for deicing or for the preparation of a heat transfer fluid is provided. The composition comprises a mixture of at least two carboxylic acid salts having a t/c ratio of 2 or lower, including a dicarboxylic salt and a monocarboxylic salt, said dicarboxylic salt being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis. More particularly, said mixture is including a succinate and a formate, wherein the succinate is in an amount of at least 50 wt %, on a dry basis. Also provided is a method for deicing a surface or preventing the accumulation of ice, snow or a mixture thereof on a surface, comprising a step of applying on a surface covered by ice, snow or a mixture thereof, or susceptible of being covered by ice, snow or a mixture thereof, the above composition. The composition is also useful for the preparation of a heat transfer fluid coolant to be used in a heat transfer system comprising a heat transfer fluid provided with a cooling system.



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DEICING AND HEAT TRANSFER FLUID COMPOSITIONS AND METHODS OF USE

FIELD OF THE INVENTION

5 [0001] The invention relates to a composition and methods of using the same in various applications such as for residential and commercial deicing applications or for the preparation of heat transfer fluids. The invention also relates to the use of heat transfer fluids so obtained. Optionally, the invention features compositions may be obtained at least in part from a fermented broth containing salts of carboxylic acid.

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BACKGROUND OF THE INVENTION

[0002] Chemical deicing is routinely used during the winter season to maintain safer conditions on sidewalks, roads, highways and airports. There are currently numerous materials and compositions used for such deicing applications. However, there are many disadvantages to the presently used materials, including their corrosiveness and their impact on the environment.

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[0003] Both acetate and formate salts, such as potassium salts, are currently used in airport runway deicing applications.^{1,2} However, there is evidence that current runway deicing products including said potassium acetate and formate cause serious threats to the integrity of the runways and the supporting infrastructure, for example at major airports. Airport managers are increasingly paying closer attention to how these chemical agents affect the integrity of runways. Any structural decay caused by such chemicals could lead to lost business and more importantly could give rise to serious safety issues. Aviation authorities believe that runway deicing products that demonstrate decreased corrosion, carbon brake oxidation, and concrete scaling will warrant a strong market positioning.

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[0004] Further, compositions with characteristics similar to those of deicing fluids are used as heat transfer fluids in numerous industrial and automotive applications and more prevalently where an operating temperature range beyond that provided by water is desired. For example, such compositions are used as heat transfer fluid compositions otherwise known as antifreeze or coolant when applied in the field of automotive engines. In motor vehicles, heat transfer fluids are used to protect engines from overheating and corrosion. However, by necessity, heat transfer fluids used in motor vehicles are antifreeze liquids to enable cold weather motor vehicle operations.

[0005] Examples of frost resistant and anticorrosion coolant compositions are disclosed in US 5,104,562. The compositions contain potassium acetate and potassium formate and may further comprise urea and ethylene glycol. In US 6,689,289 B1 compositions of monocarboxylates are used as freezing point depressants and corrosion inhibitors in heat transfer fluids. More particularly, these compositions comprise water solutions of C₁-C₂ carboxylic acid salts (e.g. potassium formate) in combination with C₃-C₅ carboxylic acid salts (e.g. sodium propanoate). Thus from the reading of this document, a person skilled in the art understands that there is synergy when combining a carboxylate with a t/c ratio of 2 or 1 with one having a t/c ratio of 3 or higher. A t/c ratio means the ratio of total carbon atoms to carboxylate carbons in the molecule (t/c ratio) is typically an important consideration. For example, acetate has two carbon atoms with one carboxylate carbon (t/c ratio of 2). Formate has one carboxylate carbon atom and one total carbon (t/c ratio of 1). Propionate has one carboxylate and three total carbons (t/c ratio of 3). A C₆-C₁₂ carboxylate may also be added (e.g. sodium heptanoate).

[0006] However, currently used coolant formulation consists of water, glycol, and small amounts of additives to minimize corrosion and foaming. The most prevalent glycol in heat transfer fluid applications is ethylene glycol occupying 98% of the market space.

[0007] Relatively small amounts of ethylene glycol can cause severe health problems or fatalities if swallowed by people or pets. The adult lethal dose is less than 7 tablespoons.³ Children can be seriously harmed with the ingestion of as little as 2 tablespoons. Health effects range from minor skin irritation to coma, respiratory failure, and death. The lethal dose for a 5 lb cat is 1 teaspoon.⁴ The lethal dose for a 22 lb dog is 1.5-2 oz.

[0008] American Association of Poison Control Centers (AAPCC) collects poisoning incident information from 64 poison centers across the United States. In the 2003 Annual Report of the AAPCC, in the category of automotive, aircraft, and boat products, 5081 human poisonings from ethylene glycol were reported with 11% of these incidents reported among children under six, and 16 cases that resulted in death.⁵

[0009] The ASPCA National Animal Poison Control Center had 510 reports of pet antifreeze poisoning with 16% deaths in a 2.5-year period. Majority of the incidents are unreported. According to AAPCC, it is generally estimated that there are 10,000 antifreeze-related deaths among pets and wildlife each year. Most pet poisonings occur in garages from faulty radiators.

[0010] Deicers, including those used in airport deicing applications, are dispensed to the surrounding environment. Similarly, about 39% of coolant is disposed of improperly, such as onto soil, into public drains and sewer systems, or into open waters.⁶ These direct releases into the environment present possible routes of exposure to human, animal, and ecological systems. Glycols such as ethylene glycol and propylene glycol used in deicing and heat transfer applications exert a high Biological Oxygen Demand (BOD) effect on receiving waters and can be detrimental to aquatic species. Biological Oxygen Demand (BOD) is the amount of oxygen required for biological oxidation by bacteria growing under aerobic conditions. Alternatives to glycols have been commercialized for airport deicing applications. However, the heat transfer fluid industries, including that catering to the motor vehicle antifreeze-coolant market,

do not have environmentally friendly, cost competitive alternatives to glycols available for consideration.

[0011] Aqueous salt solutions of succinic acid have also been proven to have deicing and heat transfer properties.^{7,8,9,10} However, such succinate based fluids have not been put to practice due to the high cost of manufacturing from petrochemical feedstocks. Biocatalytic processes such as those using fermentable sugars as a substrate are seen as an economical and environmental alternative to traditional petrochemical processes. More particularly, such processes involving conversion of low value carbohydrates, including some that are considered as waste products, are of increasing interests. For example, calcium magnesium propionate and acetate based road deicers have been produced using a fermentation process.¹¹

[0012] Micro-organisms such as *E. coli*, under anaerobic conditions, produces mixtures of carboxylic acids from fermentable broths.^{12,13} The carboxylic acids include succinic, acetic and formic acids. All commercially viable, succinate producing microorganisms described in the literature require neutralization of the fermentation broth to ensure the pH does not become too acidic or too alkaline to kill or inhibit the microbes. Neutralization of the fermentation broth results in the production of salts of succinic acid and other residual carboxylic acids such as acetic and formic.

[0013] Each of acetate, formate and succinate has positive, negative, or neutral contributory characteristics in the subject fields of use. For example, formate is highly corrosive whereas succinate has demonstrated corrosion inhibitive properties and acetate can be considered neutral. The biological oxygen demand (BOD) of formate is relatively low while BOD of succinate and acetate are higher than that of formate. In comparison to glycols used for deicing and heat transfer fluid applications, all carboxylates have a lower BOD effect and are more biodegradable.

[0014] The data presented in **Figures 1** and **2** demonstrate the benign nature of potassium succinate in comparison to acetate and formate. Dichromate treated magnesium is an alloy used in aircraft construction and is highly susceptible to corrosion in the presence of potassium formate. The effect of acetate is significant while that of succinate is substantially lower. A similar trend was observed in comparing the impact of formate, acetate, and succinate on concrete. Concrete erosion due to formate is substantial while that due to acetate is significant. The effect of succinate is negligible and is comparable to that of water.

[0015] The corrosion properties of potassium succinate and potassium acetate based heat transfer fluid, without any additives such as corrosion inhibitors, was tested and compared to a conventional ethylene glycol (EG) coolant formulation purchased from an automobile parts store. The test method used was; "Standard Test Method for Corrosion Test for Engine Coolants in Glassware," ASTM Designation: D 1384-01. The test is conducted at 70 °C under aeration to accelerate corrosion for two weeks. After two weeks, the metals are cleaned and weighed to record weight loss due to corrosion. The results are presented in the **Figure 3**.

[0016] The corrosion profile for the carboxylate based heat transfer fluid is essentially identical to that of the commercial EG coolant except for the solder specimen. Unlike the carboxylate coolant tested, commercial coolants are supplemented with corrosion inhibitors to minimize corrosion. It is anticipated that potential commercial carboxylate heat transfer fluids, including motor vehicle engine coolants, may be supplemented with corrosion and other types of inhibitors.

[0017] Compounds that are more persistent in the environment have increased opportunities of exposure to environmental receptors (plant and aquatic life forms). The oxygen demand of a compound during chemical oxidation and biological degradation is an indication of the persistence of the chemical in the environment. Chemical Oxygen Demand (COD) is the amount of oxygen required

for the chemical oxidation of compounds in water, as determined using a strong oxidant. BOD is the amount of oxygen required for biological oxidation by bacteria growing under aerobic conditions. The ratio of BOD to COD can be used to assess whether a compound is readily biodegradable. When BOD₅ is expressed as a percentage of COD, a BOD₅ that is < 1% of COD indicates a relatively non-biodegradable compound and > 10% of COD indicates a relatively degradable compound (the subscript "5" denotes a 5 day test). Biodegradability of the subject carboxylates and glycols used for deicing and heat transfer fluids are presented in **Table 1**.

10 **[0018]** Table 1: Environmental properties of runway deicing fluids (RDF) and heat transfer fluids at 50 % (wt.) concentration in water.

Properties	Potassium Succinate (g O ₂ / g fluid)	Potassium Succinate:Acetate 1:1 Ratio (g O ₂ / g fluid)	E36®* (g O ₂ / g fluid)	Potassium formate (g O ₂ / g fluid)	Ethylene Glycol (g O ₂ / g fluid)	Propylene Glycol (g O ₂ / g fluid)
BOD ₅	0.17 ¹⁴	0.20 ¹⁴	0.14 ₁₅	0.10 ¹⁶	0.4 ¹⁸	0.5 ¹⁸
COD	0.25 ¹⁴	0.28 ¹⁴	0.30 ₁₅	-	0.65 ¹⁹	0.84 ¹⁹
Biodegradability (BOD ₅ /COD) %	68 %	71 %	47 %	100 % ¹⁷	61 %	59 %

* E36® is a potassium acetate-based liquid runway deicer commercialized by the company Cryotech

15 **[0019]** Clearly, the BOD₅ of potassium formate is significantly lower than that of both acetate and succinate. However, the BOD₅ values of glycols are substantially higher than that of all the carboxylates. Therefore, glycols such as

ethylene glycol and propylene glycol used in deicing and heat transfer applications exert a high BOD effect on receiving waters, and as such can be detrimental to the environment.

[0020] The biodegradability of potassium formate, measured as a ratio of BOD:COD, is 100% in five days, which is an indication of the ease of degradation by bacteria in the environment and hence an indication that the formulation will be less persistent in the environment. Both succinate and acetate are also highly biodegradable and not considered as components that will persist in the environment. Both glycols are also readily biodegradable.

[0021] The deicing chemicals, in general, melt ice due to their tendency to form aqueous solutions that have lower freezing points. The melted or melting ice/snow is removed from pavements using mechanical devices. Similarly, heat transfer fluid chemicals function due to their tendency to form aqueous solutions that have lower freezing points and higher boiling points, effectively increasing the operating range with respect to temperature. Therefore, the freezing point of these fluids is a reasonable indicator of performance.

[0022] A comparison of the freezing points is given in **Table 2**. Although all the fluids have reasonably close freezing points, potassium acetate has a clear lead by virtue of its lower freezing point.

[0023] Table 2: Freezing point of runway deicing fluids (RDF) and heat transfer fluids at 25% (wt.) concentration in water.

Property	Potassium Succinate (°C)	Potassium Succinate:Acetate 1:1 Ratio (°C)	Potassium Acetate (°C)	Potassium formate (°C)	Ethylene Glycol (°C)	Propylene Glycol (°C)
Freezing point	-12.5 ¹⁴	-14.5 ¹⁴	-18.0 ¹⁴	-15.0 ¹⁷	-12.0 ²⁰	-11.0 ²¹

[0024] Clearly, each of the three carboxylates has distinct features that are attractive for both runway deicing and heat transfer fluid applications. Potassium succinate is, by far, the most benign deicer with respect to corrosion and structural degradation. Potassium acetate stands out as the best performing deicer and heat transfer fluid due to its characteristically low freezing point. Finally, potassium formate, having the lowest BOD, is the most ecologically sound ingredient. However, none of the carboxylates taken separately demonstrate a clear overall advantage.

[0025] Thus, there is still a need for deicing compositions (in solid or liquid form) and heat transfer fluid compositions that provide a good balance between performance and reduced corrosion and pollution attributes.

[0026] There is a need for a deicing composition (in solid or liquid form) having low corrosion and low BOD useful in deicing various surfaces such as road and runways in cold regions as well as a need for heat transfer fluid compositions having a lower BOD effect.

[0027] There is also a need for providing a biobased carboxylate salts deicing composition which is economically attractive for use as a commercial deicer, particularly at airports. A similar need exists for making biobased carboxylate salts economically attractive for use as a commercial heat transfer fluid.

[0028] The present inventors unexpectedly found that a mixture of at least two carboxylic acid salts having a t/c ratio of 2 or lower, including a dicarboxylic salt and a monocarboxylic salt, said dicarboxylic salt being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis, synergistically reduces the freezing point of aqueous solutions of the mixture. It is to be noted that it is unexpected for a person skilled in the art that a dicarboxylic salt (e.g. succinate) would show synergy with with a monocarboxylic salt (e.g. formate and/or acetate), both said dicarboxylic and monocarboxylic salts having a

t/c ratio of 2 or lower. Thus there is no need for a carboxylate having a higher t/c ratio to obtain the synergy with low monocarboxylic salts having a t/c ratio of 2 or lower.

5 [0029] More particularly, the present inventors unexpectedly found that a mixture of carboxylic salts including succinate and formate wherein said succinate is present in an amount of at least 50 wt % of said mixture, provides such compositions.

10 [0030] Optionally, according to a preferred embodiment, aforesaid composition may be obtained at least in part from a fermentation process utilizing low costs carbohydrates (including agricultural and forestry wastes or by-products). A further advantage of such compositions is that it can be made directly from a fermentation broth, which significantly reduces the costs associated with manufacturing a synthetic formulation, all the while significantly reducing or eliminating the costs for disposing agricultural and forestry wastes or byproducts.

15 SUMMARY OF THE INVENTION

Description of various embodiments of the invention

20 [0031] The present invention provides a new composition having unique and unexpected characteristics, useful in various applications such as deicing or heat transfer, and that is beneficial with respect to corrosion and the environment. Such a composition may be useful as a deicing or heat transfer fluid.

25 [0032] According to one embodiment, the invention relates to a composition for deicing or for the preparation of a heat transfer fluid, wherein said composition comprises a mixture of at least two carboxylic acid salts having a t/c ratio of 2 or lower, including a dicarboxylic salt and a monocarboxylic salt, said dicarboxylic salt being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis.

[0033] According to a particularly preferred embodiment, the invention relates to a composition for deicing or for the preparation of a heat transfer fluid, wherein said composition comprises a mixture of at least two carboxylic acid salts including a formate and a succinate, said succinate being present in the mixture in an amount of at least 50 wt % of the weight of said mixture, on a dry basis.

[0034] According to another embodiment, said mixture may be obtained at least in part from a fermentation broth comprising at least one carbohydrate source, and at least one carboxylic acid producing microorganism. Advantageously, a mixture of salts of carboxylic acids obtained at least in part by the fermentation of a carbohydrate source (sugars) in the presence of a source of nitrogen and at least one carboxylic acid producing microorganism. Optionally, the amounts of carboxylic salts may be adjusted to meet with the aforesaid proportions by mere addition and mixing of the missing portion of carboxylic salts in the mixture or in the fluid composition.

[0035] According to a preferred embodiment, the above-mentioned carboxylic acid producing microorganism may be *Aspergillus niger*, *Corynebacterium glutamicum* (also called *Brevibacterium flafum*), *Escherichia coli*, *Enterococcus faecalis*, *Veillonella parvula*, *Actinobacillus succinogenes*, *Mannheimia succiniciproducens*, *Anaerobiospirillum succiniciproducens*, *Paecilomyces varioti*, *Saccharomyces cerevisiae*, *Bacteroides fragilis*, *Bacteroides ruminicola*, *Bacteroides amylophilus*, or a mixture thereof. According to a particularly preferred embodiment, the carboxylic acid producing microorganism is *E. coli*.

[0036] According to another preferred embodiment, the carbohydrate source present in the fermentable broth which is to be used to prepare the composition of the invention comprises hexoses, pentoses or a mixture thereof. Upon fermentation in the presence of the microorganism, these hexoses, pentoses, or mixture thereof produce salts of carboxylic acids. For example, the fermentation is done according to the protocol disclosed in US Patent No. 6,743,610 B2. More preferably, the above-mentioned carbohydrate source comprises hexoses, pentoses or a mixture thereof.

[0037] According to another preferred embodiment, the mixture may further comprise additional carboxylic acid salts or catabolic organic acids. Non limitative examples of said additional carboxylic acid salts may be carboxylic acid salts of acetate, malate, fumarate, citrate, lactate, or propionate. More preferably, said additional carboxylic salt may be an acetate salt.

[0038] According to another preferred embodiment, the salts of carboxylic acid which are present in the composition of the invention are for instance potassium, sodium, ammonium, calcium, and/or magnesium salts of succinate, acetate, formate, malate, fumarate, citrate, lactate, propionate, or other catabolic organic acids. More preferably, said carboxylic acid salts may be sodium, potassium, ammonium, calcium, or magnesium salts, or mixtures thereof.

[0039] According to a particularly preferred embodiment, the above-mentioned mixture of carboxylic acid salts may comprise potassium succinate, potassium formate and potassium acetate.

[0040] In another embodiment, the fermented broth, prior to concentration, comprise up to about 200 g/L of any salts of carboxylic acid. It is worth noting that the broth may be concentrated via evaporation in order to obtain the desired carboxylate concentration. Moreover, the mixed carboxylates fermentation broth may be further treated for purification, such as for reducing color. The fermented broth may also be treated to remove the biomass and other contaminants.

[0041] In a further embodiment, the quantities of the carboxylates present in the composition of the invention may be adjusted to provide enhanced properties to the final composition. The adjustment may be done through concentrating of the fermented broth and/or by further addition of at least one of the carboxylates. For example, potassium formate can be further added to the fermented broth or concentrated in order to reduce the BOD value of the composition, thereby making the composition more attractive from an ecological stand point. The percentage of potassium formate in the composition would be of < 50 wt % in order to enhance the BOD value (the percentages being expressed in weight of the total weight of the mixture, on a dry basis). Optionally, potassium acetate or

potassium formate can be added to the fermented broth or concentrated to decrease its freezing point and enhance performance. The optimum percentage of potassium acetate or potassium formate in the composition would be < 50 wt %, each, on a dry basis, in order to decrease its freezing point. Furthermore, potassium succinate may be added to the fermented broth or concentrated in order to reduce structural damages induced by the composition such as corrosion, concrete erosion, and carbon brake oxidation. The optimum percentage of potassium succinate in the composition would be of about 65 wt %. Therefore, the present invention advantageously provides a versatile composition in which the quantities of each of the carboxylates may be adjusted in order to obtain a deicing or heat transfer fluid composition with the desired properties. The final properties of the composition may also be adjusted by balancing the quantities of each carboxylate.

[0042] According to another embodiment, the composition may comprise:

potassium succinate	50 to 90 wt%, preferably 60 to 80 wt %;
potassium formate	10 to 50 wt %, preferably 10 to 20 wt %; and
potassium acetate	0 to 40 wt%, preferably 10 to 20 wt %;

considering that the sum of the percentages is 100 wt % of the mixture, on a dry basis.

[0043] According to another embodiment, the mixture of carboxylic acid salts is in solid form. The solid mixture can be directly applied for deicing. In such a case, the mixture may be obtained by mere evaporation of the broth water and subsequent precipitation or crystallization and drying. Any appropriate means or apparatuses for evaporation, crystallization, and drying well known to those skilled in the art can be used. Alternatively, the solid mixture may be obtained by

mixing appropriate amounts in weight of carboxylic salts using appropriate means or apparatuses for mixing and blending well known to those skilled in the art.

[0044] According to another embodiment, the composition may further comprise a solvent in which the mixture of carboxylic salts is solubilized. According to a particularly preferred embodiment, the solvent may be any appropriate solvent that is not harmful to the environment, human beings or animals such as pets. Non limitative examples of solvent may comprise water, monohydric alcohols having 1 to 6 carbon atoms, polyhydric alcohols having 3 to 12 carbon atoms, monomethyl or monoethyl ethers of polyhydric alcohols having 3 to 12 carbon atoms, or mixtures thereof. More preferably, said solvent may comprise water, methanol, ethanol, propanol, isopropanol, butanol, pentanol, or mixtures thereof. Preferably, the monohydric alcohol is ethanol, methanol or an admixture of the methanol and ethanol. According to a particularly preferred embodiment, the solvent is water. Said composition comprising a solvent may be of the type ready to be used or a liquid premix requiring to be diluted before use.

[0045] According to another embodiment, the invention relates to an aqueous composition for deicing or heat transfer, wherein said composition comprises a mixture of:

potassium succinate	60 to 80 wt%;
potassium formate	10 to 20 wt %; and
potassium acetate	10 to 20 wt%;

considering that the sum of the percentages is up to 100 wt % of the mixture, and wherein said mixture is in a concentration of from 30 to 60 wt % in water. Again, this aqueous composition can be of the type ready to be used or may define a premix that can be diluted in water before use, as far as aforesaid concentrations are met.

[0046] According to another embodiment, the invention relates to a method for deicing a surface covered by ice, snow or a mixture thereof or preventing the accumulation of ice, snow or a mixture thereof on a surface, comprising a step of applying on said surface covered by ice, snow or a mixture thereof, or susceptible
5 of being covered by ice, snow or a mixture thereof, the any of the compositions defined hereinabove. The composition may be applied to the surface by any appropriate means or apparatuses well known to a person skilled in the art.

[0047] According to another embodiment, the invention relates to a use of an aqueous composition as defined hereinabove as a heat transfer fluid coolant to
10 be used in a heat transfer system comprising a heat transfer fluid provided with a cooling system.

[0048] The deicing composition of the invention is useful in any residential or commercial deicing applications. In a particular embodiment, such a surface is a runway such as an airport runway.

[0049] The present invention further concerns a method for cooling an engine comprising providing a heat transfer fluid composition of the invention as an engine coolant; introducing the composition into a cooling system of the engine;
15 and running the engine containing the coolant.

[0050] The heat transfer fluid composition of the invention is particularly useful
20 as an engine coolant for motor vehicles.

[0051] Therefore, an advantage of the compositions and methods of the present invention is that it provides a versatile composition in which the quantities of each of the carboxylates may be adjusted in order to obtain a composition with the desired properties. The final properties of the composition may be adjusted by
25 balancing the quantities of each carboxylate.

[0052] As will be shown in the examples that follow, the presence of such components in their relative quantities in the deicing or the heat transfer fluid composition has been shown to provide synergistic effect resulting in highly

efficient deicing or heat transfer/antifreeze attributes while presenting improved anti-corrosive and environmental friendly attributes.

[0053] The deicing composition of the invention is effective for deicing any residential or commercial surface. For instance, the surface to be deiced may be a runway. In a particular embodiment, the composition is used as an airport runway deicing fluid. The composition is also suitable for deicing roadways and particularly expensive structures related to roadways such as bridges, ramps, and parking facilities.

[0054] Accordingly, the present invention also provides a method of deicing a surface by application of the deicing composition of the invention. The method consists in applying to an ice/snow covered surface or applying to a bare surface prior to an ice/snow event, an amount of the deicing composition as defined hereinabove to substantially reduce the ice/snow on the surface.

[0055] The composition of the invention is useful as a heat transfer fluid composition. More particularly, the composition is useful as an engine coolant for motor vehicles such as cars, buses, trucks etc.

[0056] Thus, the present invention also provides a method for cooling an engine, comprising providing a heat transfer fluid composition of the invention as an engine coolant; introducing the composition into a cooling system of the engine; and running the engine containing the coolant.

DESCRIPTION OF THE FIGURES

[0057] Fig. 1 (prior art)¹: Dichromate treated magnesium alloy corrosion in aqueous deicer solutions containing 3 wt% deicer. Corrosion inhibitors were not used. Y = rate in mg/cm²; a = potassium formate; b = potassium acetate; c = potassium succinate.

[0058] Fig. 2 (prior art)¹: The effect of aqueous deicers on concrete. The cumulative scaled concrete, measured in grams, after 50 freeze-thaw cycles is presented. Y = amount of scaled concrete (g); a = potassium

formate; b = potassium acetate; c = potassium Succinate; d = deionized, distilled water.

5 [0059] Fig. 3 (prior art): Results for engine block corrosion test conducted according to "Standard Test Method for Corrosion Test for Engine Coolants in Glassware," ASTM Designation: D 1348-01. A = copper, B = solder, C = brass, D = steel, E = cast iron, F = cast aluminum.

10 [0060] Fig. 4: Freezing points of 25% (wt.) aqueous carboxylate solutions. The constituent relative weight composition of the solutions are provided with reference to K-ScAc:K-AcAc:K-FcAc, where K-ScAc is dipotassium succinate, K-AcAc is potassium acetate, and K-FcAc is potassium formate, and (a) = 50:0:0, (b) = 30:0:20, (c) = 25:0:25, and (d) = 0:0:50.

15 [0061] Fig. 5: Concrete scaling resulting from aqueous carboxylate solutions. The constituent relative weight composition of the solutions are provided with reference to K-ScAc:K-AcAc:K-FcAc:Water, where K-ScAc is dipotassium succinate, K-AcAc is potassium acetate, and K-FcAc is potassium formate, and (a) = 50:0:0:50, (b) = 30:20:0:50, (c) = 20:30:0:50, (d) = 10:50:0:40, and (e) = 0:50:0:50. The concrete scaling tests were conducted according to protocols specified in ASTM C 672 & C672M, "Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals."

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25 [0062] Fig. 6: Concrete scaling resulting from aqueous carboxylate solutions. The constituent relative weight composition of the solutions are provided with reference to K-ScAc:K-AcAc:K-FcAc:Water, where K-ScAc is dipotassium succinate, K-AcAc is potassium acetate, and K-FcAc is potassium formate, and (a) = 50:0:0:50, (b) = 30:0:20:50, (c) = 20:0:30:50, (d) = 10:0:50:40, and (e) = 0:0:50:50. The concrete scaling tests were conducted according to protocols specified in ASTM C 672 & C672M, "Standard Test Method for Scaling Resistance of Concrete Surfaces Exposed to Deicing Chemicals."

[0063] Fig. 7: Freezing point (°C) of carboxylate compositions of table 3 at 25 wt% in water.

[0064] Fig. 8: Freezing point (°C) of binary carboxylate compositions of table 3 at 25 wt % in water.

5 [0065] Fig. 9: Concrete Scaling (kg/m²) of 50 wt% aqueous carboxylate compositions of table 3 tested per ASTM C 672 & C672M.

[0066] Fig. 10: Concrete Scaling (kg/m²) of 50 wt% aqueous binary carboxylate compositions of table 3 tested per ASTM C 672 & C672M.

DETAILED DESCRIPTION OF THE INVENTION

10 [0067] The present invention provides a novel composition having unexpected properties for deicing or for the preparation of heat transfer fluid, wherein said composition comprises a mixture of at least two carboxylic acid salts having a t/c ratio of 2 or lower, including a dicarboxylic salt and a monocarboxylic salt, said dicarboxylic salt being present in the mixture in an amount of at least 50 wt % of
15 the weight of the mixture, on a dry basis. More preferably, the mixture may comprises at least two carboxylic acid salts including a formate and a succinate, said succinate being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis. Optionally, said mixture may be obtained at least in part from a fermented broth.

20 [0068] The invention also provides a method of deicing a runway surface by application of the deicing composition of the invention. The invention further concerns a method for cooling an engine using the heat transfer fluid composition of the invention as an engine coolant. The invention further concerns a method for heat transfer using the heat transfer fluid composition of the invention in
25 industrial applications. Moreover, the present invention provides a method for producing a composition having enhanced characteristics by providing a base composition and adjusting the quantities of each of the carboxylates in order to obtain the composition with the desired characteristics.

Definitions

[0069] The ratio t/c means the ratio of total carbon atom to carboxylic groups. For example, acetate has two carbon atoms with one carboxylate carbon (t/c ratio of 2). Formate has one carboxylate carbon atom and one total carbon (t/c ratio of 1). Propionate has one carboxylate and three total carbons (t/c ratio of 3).

[0070] The expression **fermented broth** or **fermented broth mixture** according to the invention refers to a broth containing at least one carboxylic acid salt, obtained by fermentation of a fermentable broth comprising one or more carbohydrates or sugars in the presence of a source of nitrogen and at least one carboxylic acid producing organism. For fermentation technologies targeted for chemical industries that are typically classified as "high volume/low value" processes, the fermentable broth can be formulated using inexpensive agricultural and forestry waste/byproducts such as corn steep liquor/solids, which contain nutrients in numerous and significant proportions. Some elemental and nutritional fortification of the media using small amounts of inorganic salts and nutrients may be necessary to satisfy physiological requirements of specific microorganisms. Generally, the most productive and economical combination that will satisfy requirements for cell biomass and metabolite production, energy requirements, as well as fermentability requirements are considered in formulating the fermentable broth. Carbohydrates utilized in fermentable broths are numerous. Conventional carbohydrates include glucose, fructose, and sucrose. The latter is a disaccharide glucoside, which is utilized in a number of fermentation processes including the production of proteins, ethanol, organic acids, and amino acids. Hydrolyzed structural polysaccharides from plant biomass are considered as next generation substrates for fermentable broths. Hydrolysis of cellulose and hemicelluloses provide several hexoses (glucose and mannose) and pentoses (xylose and arabinose) for fermentation. Batch fermentations may utilize in excess of 100 g/L of substrate and continuous or fed-batch fermentation may utilize 0.5 – 4.0 g/L/hr of substrate.

[0071] A **carboxylic acid producing organism** according to the invention means an organism capable of producing a carboxylic acid from a carbohydrate source. For example, the organism may be one or a mixture of *Aspergillus niger*, *Corynebacterium glutamicum* (also called *Brevibacterium flafum*), *Escherichia coli*, *Enterococcus faecalis*, *Veillonella parvula*, *Actinobacillus succinogenes*,
5 *Mannheimia succiniciproducens*, *Anaerobiospirillum succiniciproducens*, *Paecilomyces varioti*, *Saccharomyces cerevisiae*, *Bacteroides fragilis*, *Bacteroides ruminicola*, *Bacteroides amylophilus*, or any other organism capable of producing carboxylic acids. In one embodiment, the organism is the
10 microorganism *E. coli*.

[0072] The **carboxylic acid salt** according the invention is a salt of a carboxylic acid produced by a microorganism by fermentation of carbohydrates contained in a fermentable broth. The carboxylic acid salt may be for instance a monocarboxylic acid, a dicarboxylic acid salt, a tricarboxylic acid or a mixture
15 thereof. According to a particular embodiment, the invention encompasses a mixture of such carboxylic acid salts. For example, the carboxylic acid salts are potassium, sodium, ammonium, calcium, and/or magnesium salts of succinate, acetate, formate, malate, fumarate, citrate, lactate, propionate, or other catabolic organic acids, or mixture thereof. According to a particular embodiment, the
20 composition of the invention comprises a mixture of succinate, acetate and formate. Even more preferably, the composition comprises a mixture of potassium succinate, potassium acetate and potassium formate.

[0073] The expression **catabolic organic acids** used in the context of the invention means organic acids found in fermented broths resulting from the
25 metabolism of microorganism used in fermentation processes.

[0074] The term **amount** used in the context of the use of invention represents an amount of the deicing composition of the invention which is necessary to reduce the quantity of ice and/or snow present on the surface to be deiced. In a particular embodiment, such amount allows reduction or melting of ice and/or
30 snow so that safe conditions are restored allowing the surface to be used for

normal activities. Deicing can be accomplished by application of the deicer either prior to the icing/snowing event or following the icing/snowing event.

[0075] A **heat transfer fluid composition** used in the context of the invention means a fluid composition having good heat transfer properties, particularly for cooling an engine during use, while also having antifreeze properties to prevent freezing when the engine is not active in cold weather.

[0076] The expression **enhanced characteristics** used in the context of the present invention refers to the characteristics which are intended to be present in the composition. These enhanced characteristics will depend on the particular application which is intended for the composition. For instance, the enhanced characteristic can be an enhanced ecological property. It can also be an enhanced anticorrosive characteristic or an enhanced antifreeze property. The composition may also have a combination of these enhanced characteristics. A composition having enhanced characteristics according to the invention may be a composition which has good ecological and/or anticorrosion and/or antifreeze characteristics required for a specific use.

EXAMPLE:

[0077] In the context of a deicing composition for use on airport runways and other surfaces that require deicing, the desired characteristics of the composition are well known. The characteristics include good performance with respect to ice melting, low impact on structural components including aircraft alloys, steel, and concrete, and low BOD to minimize impact on the environment. The data presented above clearly demonstrate that succinate based deicer formulations can have a substantial effect on lowering the impact on both structural components such as aircraft alloys, steel, and concrete and the environment.

[0078] The freezing point of deicer solutions is the primary indicator for the performance of deicers. It is also an important characteristic of heat transfer fluids since they are typically formulated to function in cold regions. The freezing point can be measured at both 50% and 25%. In the runway deicer industry, it is

measured at 25% due to dilution of the deicer upon application as well as ease of measurement; i.e., the 50% commercial solution is diluted to 25% prior to measurement. The critical value for a freezing point of the 1:1 dilution of a commercial deicer with water should be less than -14.5 deg. C for airport runway deicing applications. Typically, a commercial solution is 50%. It has been discovered that mixed carboxylate salt solutions, particularly those that can be derived from fermented broths, demonstrate synergistic enhancement of freezing points, indicating that such solutions would provide enhanced performance compared to that of the individual carboxylates. The data are summarized in Table 3 and Figures 4, 7 and 8.

[0079] Airport and Airplane deicers are required to be compliant with stringent requirements set forth by AMS 1435 A.²² The document specifies a series of tests for deicers including: (1) Freezing point (ASTM D 1177); and (2) Runway concrete scaling resistance (ASTM C 672). These protocols were followed to obtain the data presented in Table 3 and Figures 4, 5, and 6.

[0080] Figure 4 demonstrates the synergistic enhancement in freezing point of mixed dipotassium succinate and potassium formate solutions. The composition consisting of equal amounts of both carboxylate salts show a noticeably lower freezing point than that of potassium formate. Further, the succinate:formate = 30:20 composition, which contains less formate, shows a freezing point that is slightly lower than the freezing point of potassium formate.

[0081] The effect of potassium formate on the freezing points of potassium carboxylate mixtures can be observed in data presented in Table 3 and in Figures 7 and 8. The freezing point of the succinate:acetate:formate = 40:10:0 composition (Test No. 4) was observed to be -13 °C. However, the substitution of half of the acetate content with formate resulting in the succinate:acetate:formate = 40:5:5 composition (Test No. 5) results in a freezing point of -16 °C. This was a

substantial enhancement of the freezing point in spite of the quantitatively equivalent substitution. A similar enhancement was observed with Test No. 8 and 9. The freezing point of the succinate:acetate:formate = 30:20:0 composition (Test No. 9) was observed to be -14 °C. The substitution of half of the acetate content with formate resulting in the succinate:acetate:formate = 30:10:10 composition (Test No. 8) results in a freezing point of -19 °C. The data suggests that the preferred deicer compositions are those that consist of a relatively greater amount of succinate compared to the sum of acetate and formate where the sum of acetate and formate is made up of equal amounts of acetate and formate; for example, compositions presented in Test No. 5 and 8.

[0082] Previously it had been demonstrated that potassium succinate has a very low impact on concrete compared to the high impact from both potassium acetate and potassium formate (Figure 2). The benign nature of potassium succinate is confirmed in the data presented in Table 3 and Figures 9 and 10. The data indicate that potassium acetate (Test No. 2) is 5360% more harmful on concrete relative to potassium succinate (Test No. 1) and potassium formate (Test No. 3) is 14,840% more harmful on concrete relative to potassium succinate (Test No. 1).

[0083] During the present effort, it was discovered that the mixing of potassium succinate with both potassium acetate and potassium formate leads to a disproportionate reduction in the degree of concrete scaling. The data are presented in Figure 5 and Figure 6. Figure 5 shows that a 16.6 % substitution of potassium acetate with potassium succinate (deicer solution (d)) leads to a 98.2% reduction in concrete scaling. The data demonstrate that the substitution of potassium acetate with potassium succinate in all proportions leads to a highly disproportionate reduction in concrete scaling, which is not predicated by prior art. The prior art predicated that the reduction in concrete scaling due to substitution of potassium acetate with potassium succinate would be proportionate to the rate of substitution. That is, a 20% substitution would lead to a 20% reduction in concrete scaling. Clearly, the discovery of the highly

disproportionate positive impact of potassium succinate is novel and valuable with respect to formulating aqueous carboxylate compositions for deicing application.

[0084] Figure 6 shows a similar effect for substitution of potassium formate with potassium succinate. A 16.6 % substitution of potassium formate with potassium succinate (deicer solution (d)) leads to a 63.5% reduction in concrete scaling. A 40% substitution of potassium formate with potassium succinate (deicer solution (c)) leads to a 84.3% reduction in concrete scaling. Although the relative effect in the case of succinate and formate mixtures are not as pronounced as that observed with succinate and acetate mixtures, the effect is substantial, novel, and valuable to the deicing industry.

[0085] Above, it was argued that the preferred deicer compositions, predicated based on freezing point data, are those that consist of a relatively greater amount of succinate compared to the sum of acetate and formate where the sum of acetate and formate is made up of equal amounts of acetate and formate; for example, compositions presented in Test No. 5 and 8. The concrete scaling data also show that such compositions lead to very low concrete scaling, suggesting that those compositions have a better overall profile with respect to performance and impact on infrastructure.

[0086] As discussed above, dipotassium succinate is, by far, the most benign deicer with respect to corrosion and structural degradation. The current data also suggest that the inclusion of dipotassium succinate in potassium carboxylate mixtures leads to synergistic enhancement of the freezing point of the mixtures and therefore performance as well as a highly disproportionate reduction in concrete scaling. In combination, the discovered compositions will provide deicing and heat transfer fluid compositions with enhanced performance and reduced corrosion, concrete scaling, and pollution attributes.

[0087] Table 3: Experimental data on freezing point and concrete scaling of potassium carboxylate solutions. The data for freezing point and concrete scaling were collected according to the protocols given in ASTM D 1177 and ASTM C

672 / C 672 M, respectively. K-ScAc = potassium succinate, K-AcAc = potassium acetate, and K-FcAc = potassium formate

Test No.	Composition of Carboxylates % (wt.) in Aqueous Solution			Freezing point at 50% Dilution °C	Concrete Scaling Kg/m ²
	K-ScAc	K-AcAc	K-FcAc		
1	50	0	0	-12	0.05
2	0	50	0	-18	2.83
3	0	0	50	-15	7.47
4	40	10	0	-13	-
5	40	5	5	-16	0.05
6	35	5	10	-17	-
7	35	10	5	-17	-
8	30	10	10	--19	0.29
9	30	20	0	-14	0.05
10	30	0	20	-17	0.49
11	25	25	0	-15	-
12	25	0	25	-21	-
13	20	15	15	-17	0.73
14	20	30	0	-15	0.05
15	20	0	30	-	1.17
16	10	50	0	-	0.05
17	10	0	50	-	2.73

- [0088]** The present invention has been described with regard to preferred
 5 embodiments. The description as much as the drawings were intended to help
 the understanding of the invention, rather than to limit its scope. It will be
 apparent to one skilled in the art that various modifications may be made to the
 invention without departing from the scope of the invention as described herein,
 and such modifications are intended to be covered by the present description.
 10 The invention is defined by the claims that follow.

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CLAIMS

1. A composition for deicing or for the preparation of a heat transfer fluid, wherein said composition comprises a mixture of at least two carboxylic acid salts
5 having a t/c ratio of 2 or lower, including a dicarboxylic salt and a monocarboxylic salt, said dicarboxylic salt being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis.
2. A composition for deicing or for the preparation of a heat transfer fluid, wherein said composition comprises a mixture of at least two carboxylic acid salts
10 including a formate and a succinate, said succinate being present in the mixture in an amount of at least 50 wt % of the weight of the mixture, on a dry basis.
3. The composition of claim 2, wherein said mixture is obtained at least in part from a fermentation broth comprising at least one carbohydrate source, and at least one carboxylic acid producing microorganism.
- 15 4. The composition according to claim 3, wherein the carboxylic acid producing microorganism is *Aspergillus niger*, *Corynebacterium glutamicum*, *Escherichia coli*, *Enterococcus faecalis*, *Veillonella parvula*, *Actinobacillus succinogenes*, *Mannheimia succiniciproducens*, *Anaerobiospirillum succiniciproducens*, *Paecilomyces varioti*, *Saccharomyces cerevisiae*, *Bacteroides fragilis*,
20 *Bacteroides ruminicola*, *Bacteroides amylophilus*, or a mixture thereof.
5. The composition according to claim 2, wherein carboxylic acid producing microorganism is *E. coli*.
6. The composition according to any one of claims 3 to 5, wherein the carbohydrate source comprises hexoses, pentoses or a mixture thereof.
- 25 7. The composition according to any one of claims 1 to 6, wherein it further comprises additional carboxylic acid salts of catabolic organic acids.

8. The composition according to any one of claims 1 to 6, wherein it further comprises additional carboxylic acid salts of acetate, malate, fumarate, citrate, lactate, or propionate.

9. The composition according to any one of claims 1 to 6, wherein it further
5 comprises an acetate salt.

10. The composition according to any one of claims 1 to 9, wherein said carboxylic acid salts are sodium, potassium, ammonium, calcium, or magnesium salts, or mixtures thereof.

11. The composition according to any one of claim 1 to 10, wherein the mixture
10 of carboxylic acid salts comprises potassium succinate, potassium formate and potassium acetate.

12. The composition according to claim 11, wherein it comprises a mixture of:

potassium succinate	50 to 90 wt%,
potassium formate	10 to 50 wt %,
potassium acetate	0 to 40 wt%,

15 considering that the sum of the percentages is 100 wt % of said mixture, on a dry basis.

13. The composition according to claim 12, wherein it comprises a mixture of:

potassium succinate	60 to 80 wt%,
potassium formate	10 to 20 wt %,
potassium acetate	10 to 20 wt%,

considering that the sum of the percentages is 100 wt % of said mixture, on a dry basis.

14. The composition according to any one of claims 1 to 13, wherein carboxylic acid salts are in solid form.
15. The composition according to any one of claims 1 to 13, wherein it further comprises a solvent in which said mixture of carboxylic salts is solubilized.
- 5 16. The composition according to claim 15, wherein the solvent is water.
17. An aqueous composition for deicing or for the preparation of a heat transfer fluid, wherein said composition comprises a mixture of:
- | | |
|---------------------|----------------|
| potassium succinate | 60 to 80 wt%, |
| potassium formate | 10 to 20 wt %, |
| potassium acetate | 10 to 20 wt%, |
- considering that the sum of the percentages is 100 wt % of said mixture, and wherein said mixture is in a concentration of from 30 to 60 wt % in water.
- 10 18. A method for deicing a surface or preventing the accumulation of ice, snow or a mixture thereof on a surface, comprising a step of applying on a surface covered by ice, snow or a mixture thereof, or susceptible of being covered by ice, snow or a mixture thereof, the composition defined in any one of claims 1 to 17.
- 15 19. Use of the composition defined in any one of claims 1 to 17 for the preparation of a heat transfer fluid coolant to be used in a heat transfer system comprising a heat transfer fluid provided with a cooling system.

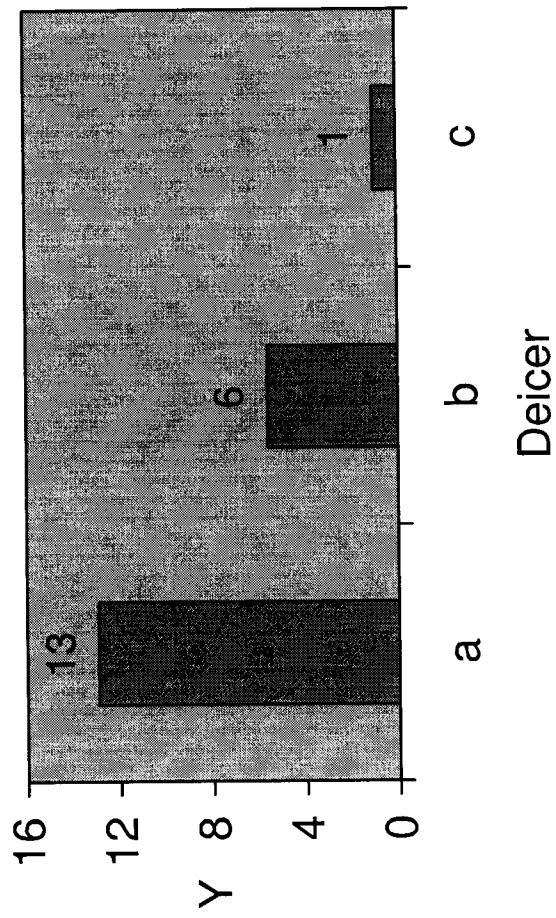


Fig. 1 (prior art)

2/10

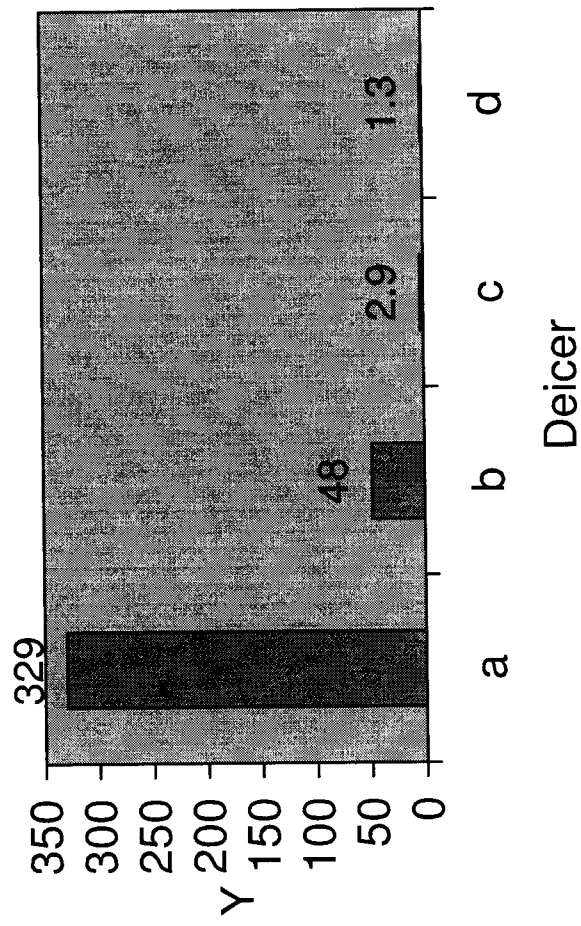


Fig. 2 (prior art)

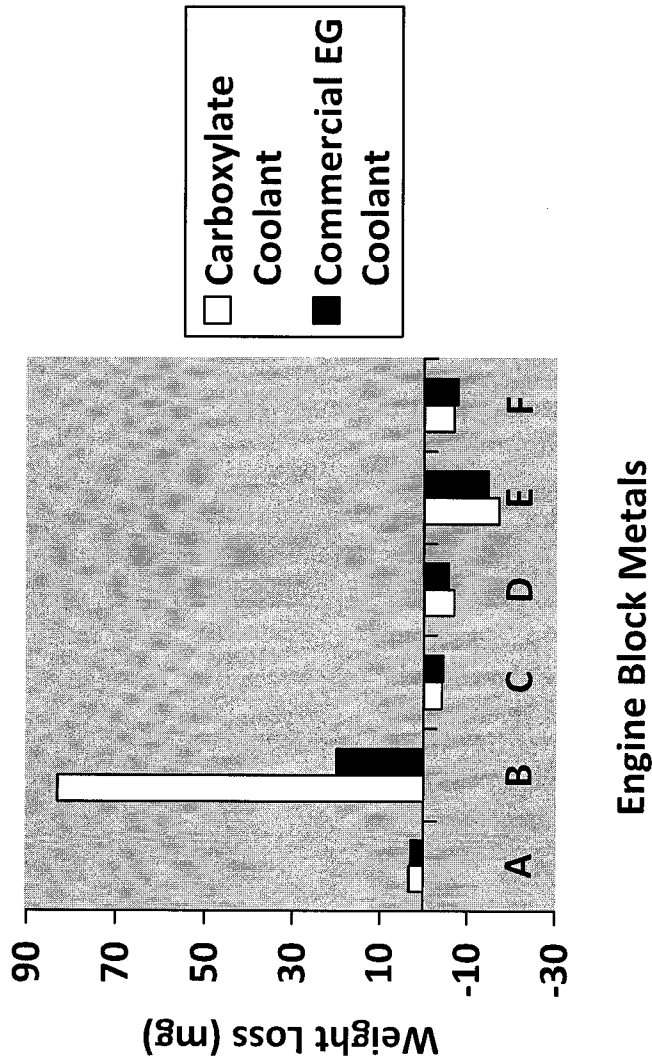


Fig. 3 (prior art)

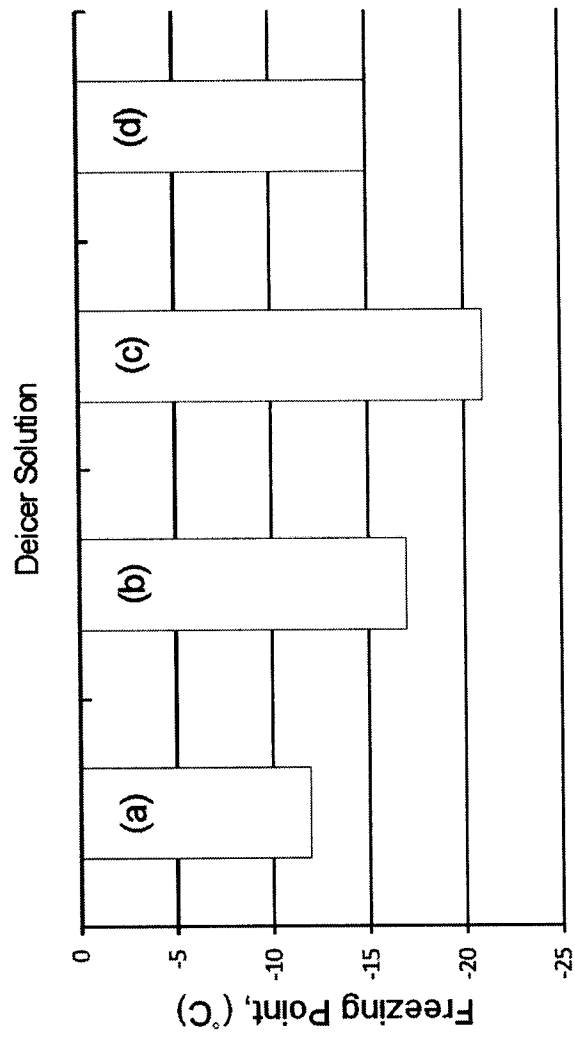


Fig. 4

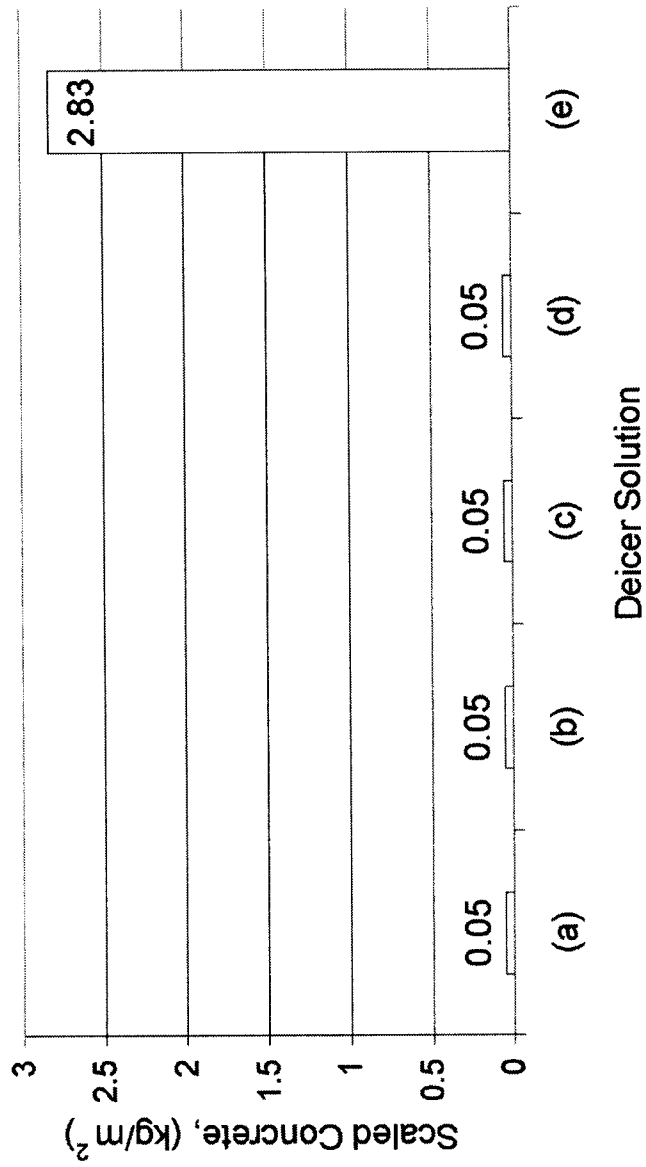


Fig. 5

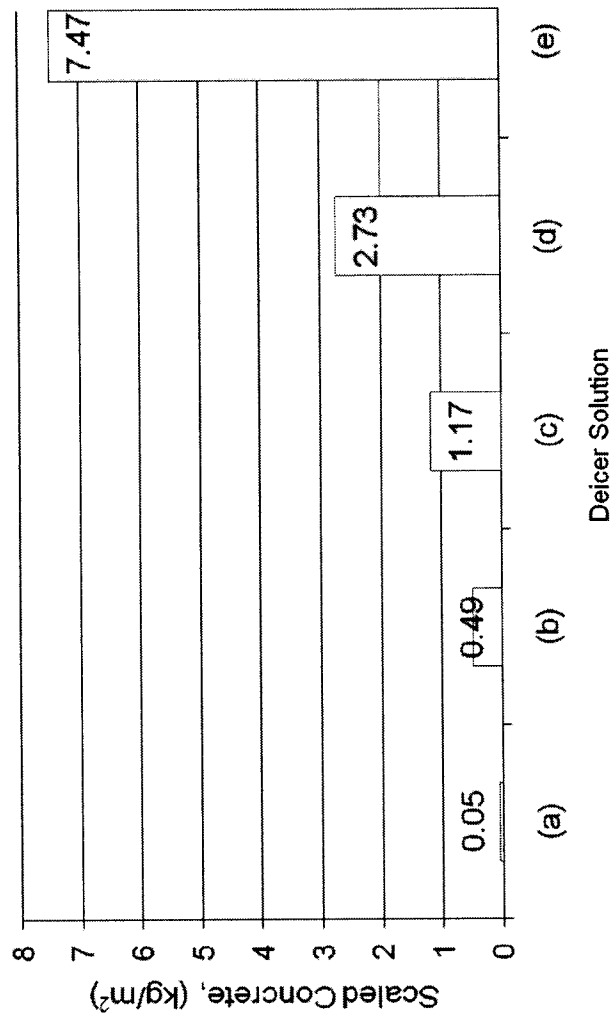


Fig. 6

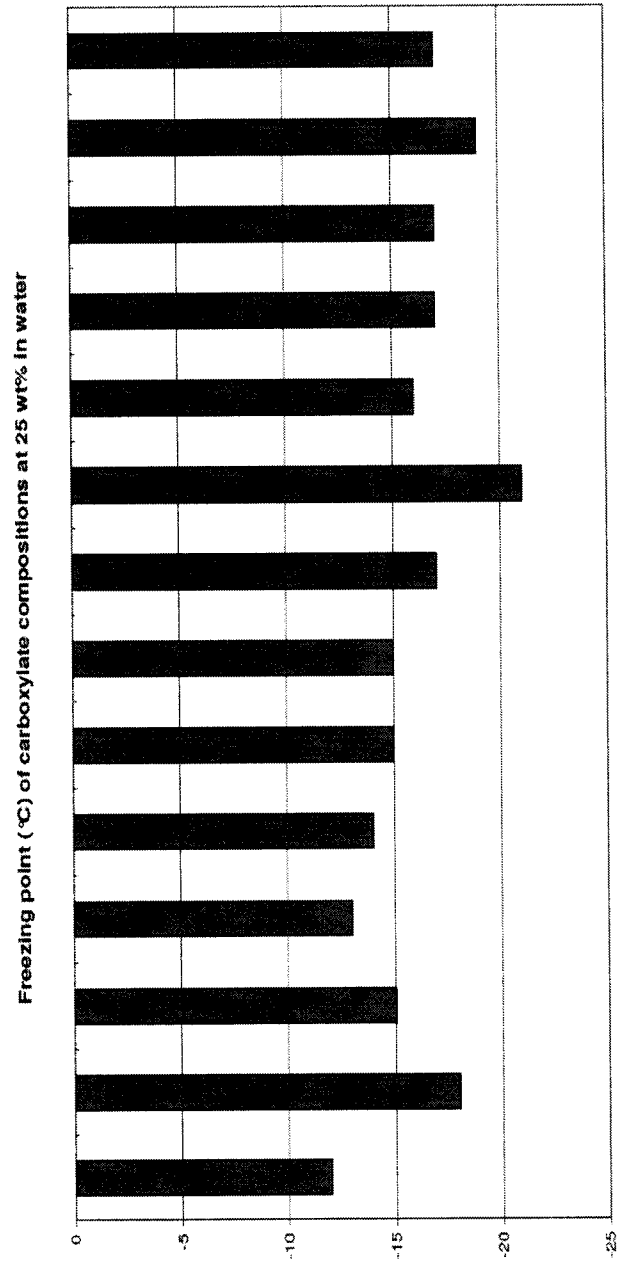


Fig. 7

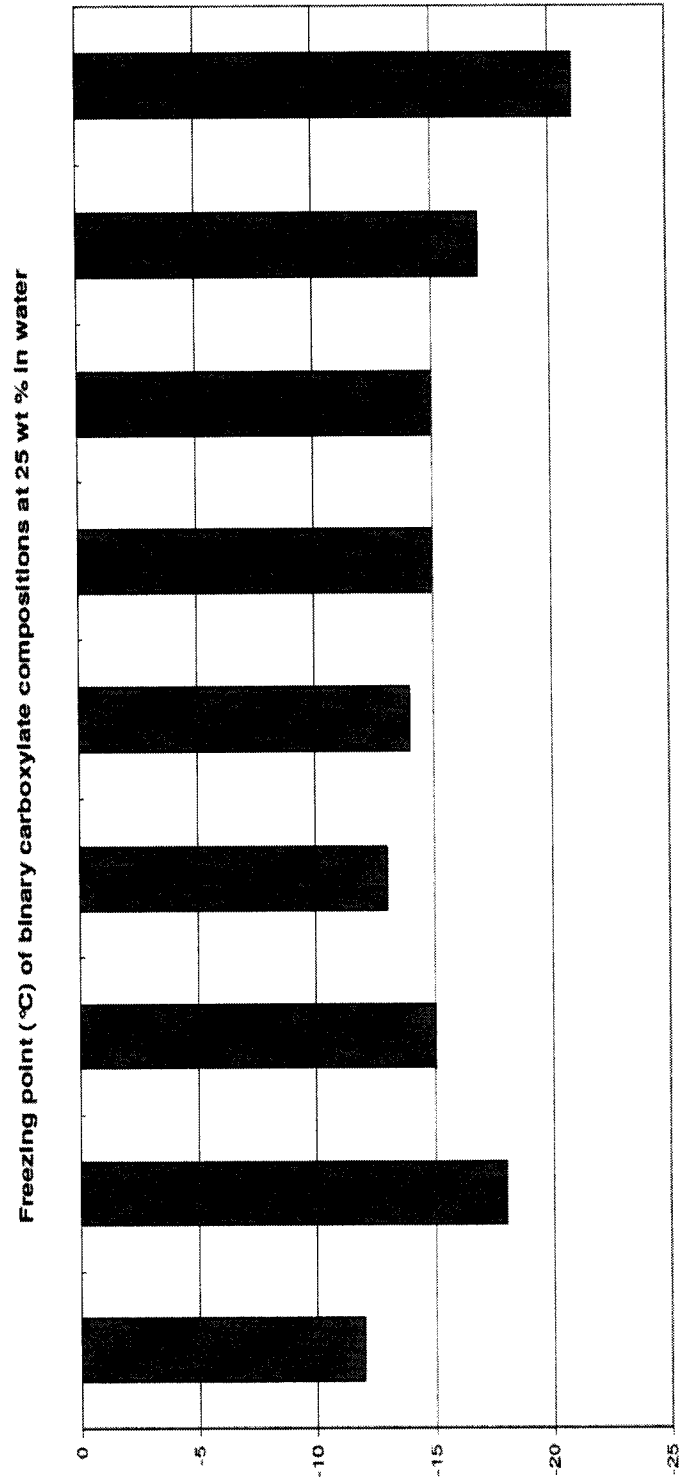


Fig. 8

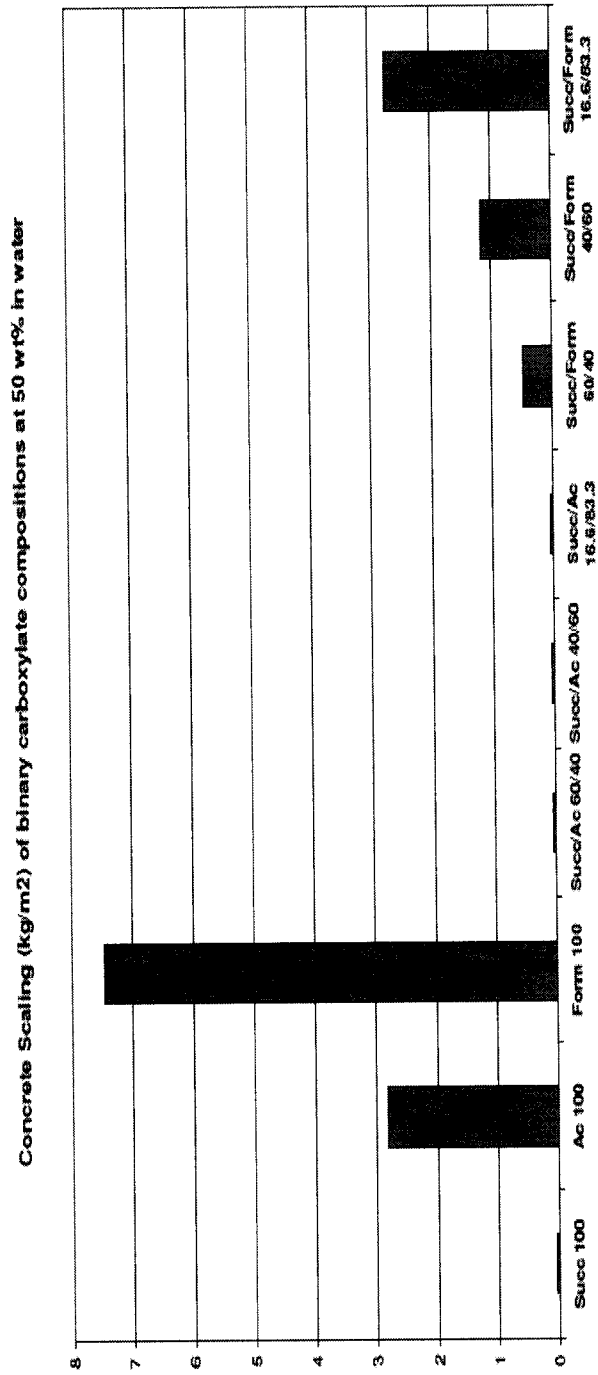


Fig. 10

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CA2009/000809

<p>A. CLASSIFICATION OF SUBJECT MATTER IPC: <i>C09K 5/10</i> (2006.01) , <i>C09K 3/18</i> (2006.01) , <i>C12P 7/40</i> (2006.01) , <i>C12P 7/46</i> (2006.01) , <i>F01P 3/00</i> (2006.01) , <i>C09K 5/20</i> (2006.01) According to International Patent Classification (IPC) or to both national classification and IPC</p>		
<p>B. FIELDS SEARCHED</p>		
<p>Minimum documentation searched (classification system followed by classification symbols) IPC(2006.01): C09K, C12P, F01P USPC: 252</p>		
<p>Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched</p>		
<p>Electronic database(s) consulted during the international search (name of database(s) and, where practicable, search terms used) Canadian Patents Database, Delphion (all databases), USPTO , QPat Keywords: succinate, succinic, butanedioic, deicer, antifreeze, coolant, heat transfer fluid</p>		
<p>C. DOCUMENTS CONSIDERED TO BE RELEVANT</p>		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	CA 2 601 759 A1 (SAPIENZA, R. et al.) 02 August 2007 (02-08-2007) * claims 1 - 3, 7, 10, 11, 19, 21, 28, pages 7, 14 - 15 *	1 - 4, 6 - 19 5
X Y	US 6,846,431 B1 (DUNUWILA, D. et al.) 25 January 2005 (25-01-2005) * cited by applicant * * claims 1 - 4, column 4, lines 36 - 59, Example 1 *	1, 8, 10, 15, 16, 19 5
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A	CA 2 318 258 A1 (STARZMANN, M.) 29 July 1999 (29-07-1999) * entire document *	1, 2, 7 - 19
<p><input type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.</p>		
*	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
“E”	earlier application or patent but published on or after the international filing date	“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
“L”	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	“&” document member of the same patent family
“O”	document referring to an oral disclosure, use, exhibition or other means	
“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		Date of mailing of the international search report
08 July 2009 (08-07-2009)		4 September 2009 (04-09-2009)
Name and mailing address of the ISA/CA Canadian Intellectual Property Office Place du Portage I, C114 - 1st Floor, Box PCT 50 Victoria Street Gatineau, Quebec K1A 0C9 Facsimile No.: 001-819-953-2476		Authorized officer Patrick Koch 819- 997-2835

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CA2009/000809

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