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(54) Title: LOW FREEZING ELECTROLYTE FOR AN ELECTROLYTIC CAPACITOR

(57) Abstract: An electrolytic capacitor comprising an anode, cathode and an electrolyte. The electrolyte comprises: about 35 to 60%, by weight, water; about 10 to 55%, by weight, organic solvent; about 0.05 to 10%, by weight, sulphuric acid; about 0.05 to 10%, by weight, boric acid; and about 0.05 to 10%, by weight, phosphorus oxy acid. The organic solvent excludes ethylene glycol, formamide, dimethyl formamide and 8-butyrolactone, and is selected from a group consisting of glycerol 1,3 propane diol; 2 methyl 1,3 propane diol, propylene glycol; polyethylene glycol monomethyl ether; N alkyl 2 pyrrolidone and diethylene glycol.



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TITLE**LOW FREEZING ELECTROLYTE FOR AN ELECTROLYTIC CAPACITOR****BACKGROUND OF THE INVENTION**

[0001] The present invention relates to an electrolyte for an electrolytic capacitor
5 and a capacitor formed therewith.

[0002] There has been an ongoing demand for ever smaller electrical components to support the continual drive to smaller devices. Of particular interest for the present invention is the demand for smaller, yet higher capacitance density, electrolytic capacitors. This demand has exhausted the current technical capabilities thereby
10 requiring further advances in the art. Such an advance is provided herein.

[0003] Electrolytic capacitors, particularly tantalum-based capacitors, have been prepared utilizing aqueous solutions of ethylene glycol with ionogens such as acetic acid and phosphoric acid and ammonium acetate. Capacitors of this type are exemplified in U.S. Patent No. 6,219,222. While these capacitors have historically fulfilled many of
15 the necessary requirements they are deficient. Other solvents are desirable that eliminate sparking and that improve resistance to electrochemical degradation.

[0004] Yet another problem associated with prior art working electrodes is the degradation of the electrolyte that occurs at low temperature. Prior art capacitors must be maintained at temperatures above the freezing point of the electrolyte. This has led
20 away from the use of aqueous electrolytes while relinquishing the superior solubility properties and charge carrying properties of water. Water is greatly preferred over organic solvents yet the propensity for freezing is detrimental to its use as a solvent for working electrolytes.

The present invention is directed to an aqueous electrolyte, and capacitor containing
25 the electrolyte, which mitigates the deficiencies of the prior art.

BRIEF SUMMARY OF THE INVENTION

[0005] It is an object of the present invention to provide an electrolytic capacitor which has a high breakdown voltage (150-300V) with suitable conductivity.

[0006] It is another object of the present invention to provide a working
30 electrolyte, and capacitor activated by the electrolyte, which can withstand low temperatures, below about -30°C without detriment to the capacitor or its properties.

[0007] A particular feature of the present invention is the ability to take

advantage of the solubilizing and conduction properties of water while still providing a capacitor able to withstand temperature storage, or use, of at least -30°C at about one atmosphere pressure.

[0008] These and other advantages, as would be realized to one of ordinary skill
5 in the art, are provided in an electrolytic capacitor. The capacitor comprises an anode, cathode and an electrolyte. The electrolyte comprises about 35-60%, by weight water; about 10-55%, by weight organic solvent; about 0.05 to 10%, by weight, sulphuric acid; about 0.05 to 10%, by weight, boric acid; and about 0.05 to 10%, by weight, phosphorus oxy acid.

10 [0009] Another embodiment is provided in an electrolyte for activating a capacitor. The electrolyte comprises about 35-60%, by weight water; about 10-55%, by weight organic solvent; about 0.05 to 10%, by weight, sulphuric acid; about 0.05 to 10%, by weight, boric acid; and about 0.05 to 10%, by weight, phosphorus oxy acid.

[00010] Another embodiment is provided in a method for providing a capacitor
15 comprising the steps of:

- a) providing a tantalum anode;
- b) providing a cathode;
- c) activating the anode and the cathode with an electrolyte comprising: about 35-60%, by weight water; about 10-55%, by weight organic solvent; about 0.05 to 10%, by
20 weight, sulphuric acid; about 0.05 to 10%, by weight, boric acid; and about 0.05 to 10%, by weight, phosphorus oxy acid; wherein the electrolyte has a pH below about 5 and a freezing point below about -30°C .

DETAILED DESCRIPTION OF THE INVENTION

[00011] The inventors have developed through diligent research an electrolyte,
25 particularly suitable for use in electrolytic capacitors, comprising water, organic solvent and acids wherein the freezing point of the aqueous electrolyte is below -30°C .

[00012] Electrolytic capacitors, particularly tantalum electrolytic capacitors, fabricated so as to manifest a large capacitance density (i.e., high capacitance per unit volume) at intermediate use voltages (i.e., from about 175 volts to about 250 volts) are
30 generally fabricated from powder metallurgy compacts. The compacts are sintered at appropriately high temperature and are then anodized in an anodizing electrolyte prior to assembly into finished capacitors. During the assembly operation each anode compact is

impregnated with a working or fill electrolyte which serves to conduct current from the cathode surface of the device to the anode oxide dielectric. Once the anode body is impregnated with a working electrolyte, the device is sealed so as to prevent escape of the liquid electrolyte. The device is typically tested prior to being placed into service.

5 The working electrolyte usually is characterized as having a much lower resistance and dissipation factor than anodizing electrolytes. One undesirable consequence of the relatively low electrical resistivity of the working electrolyte is that the breakdown voltage of the electrolyte, that is the maximum voltage which the electrolyte-anode system will support regardless of the voltage to which the anode is anodized, is generally
10 significantly lower than that of appropriate anodizing electrolytes. Working electrolytes have to be chosen so as to have a sufficiently high breakdown voltage so as not to cause premature failure during the working life of the device.

[00013] The aqueous working electrolyte solution of the present invention comprises about 35 to about 65%, by weight, water; about 10 to about 55%, by weight
15 organic solvent; about 0.05 to about 10%, by weight, sulphuric acid, about 0.05 to about 10%, by weight, boric acid and about 0.05 to about 10%, by weight, phosphorus oxy acid.

[00014] More preferably the aqueous electrolyte comprises about 40 to about 60%, by weight, water and about 35 to about 55%, by weight, organic solvent; about 1 to 5%,
20 by weight, sulphuric acid, about 1 to 5%, by weight, boric acid and about 1 to 5%, by weight, phosphorus oxy acid.

[00015] An alternate preferred aqueous electrolyte comprises about 40 to about 60%, by weight, water and about 35 to about 55%, by weight, organic solvent; and about 1 to 30%, by a mixture of ammonium acetate, acetic acid, boric acid and phosphorus
25 oxy acid.

[00016] More preferably the aqueous electrolyte comprises about 40 to about 60%, by weight, water and about 35 to about 55%, by weight, organic solvent; about 1 to 5%, by weight, boric acid and about 1 to 5%, by weight, phosphorus oxy acid and about 1 to 5%, by weight, ammonium acetate with a sufficient amount of acetic acid to adjust pH.

30 [00017] The organic solvent is preferably selected from a group consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene glycol; polyethylene glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol. The organic

solvent specifically excludes ethylene glycol, formamide, dimethyl formamide and γ -butyrolactone.

[00018] Tantalum is the most preferred anode.

[00019] The cathode is a conductive metal provided with a semiconductive or
5 metal-like conductive coating. The coating can be an oxide, nitride, carbide, or
activated carbon. Suitable cathode metals include tantalum, iridium, platinum,
palladium, gold, silver, ruthenium, tungsten, rhodium, vanadium and osmium. A
particularly preferred cathode electrode comprises a porous ruthenium oxide film
provided on a titanium substrate.

10 [00020] It is preferable that the electrolyte be maintained at an acidic pH. More
preferred is a working electrolyte with a pH of below about 5. A pH of below about 4 is
most preferred.

[00021] The pH is adjusted by balancing the sulphuric, boric and phosphorus oxy
acid and ammonium hydroxide. Suitable phosphorus oxy acids are orthophosphoric
15 acid and phosphorous acid. A particular advantage of the present invention is the
observation that the combination of acids, as set forth herein, provides a suitably high
breakdown voltage with an electrolyte solution which has a suitably high conduction and
a low freezing point.

[00022] An optional, but preferred, separator material physically separates the
20 anode and cathode from each other. The separator prevents electrical short circuits
between the anode and cathode. The separator material is preferably unreactive with the
anode, cathode and electrolyte and is sufficiently porous to allow the electrolyte to freely
flow through the separator. Suitable separators include woven and non-woven fabrics of
polyolefinic fibers, as known in the art.

25 [00023] A comparative electrolyte as described in Example 1 of U.S. Patent No.
6,219,222 would be prepared. An inventive aqueous electrolyte was prepared
comprising about 36% glycerol, about 8%, by weight ammonium acetate, about 8%, by
weight, acetic acid and about 0.5%, by weight, phosphoric acid. The breakdown voltage
was determined to be about 250 to 275 volts. The results were achieved without
30 ethylene glycol and therefore the problems associated with the detrimental side products
of the electrochemical oxidation of ethylene glycol are eliminated. These products,
including formic and oxalic acid, decrease the sparking voltage with time. The decrease

in sparking voltage is eliminated with the present invention.

[00024] The invention has been described with particular emphasis on the preferred embodiments. It would be realized from the teachings herein that other embodiments, alterations, and configurations could be employed without departing from
5 the scope of the invention which is more specifically set forth in the claims which are appended hereto.

Claims

What is claimed is:

1. An electrolytic capacitor comprising an anode, cathode and an electrolyte, said electrolyte further comprising:
 - 5 about 35-60%, by weight water;
 - about 10-55%, by weight organic solvent with the proviso that said organic solvent excludes ethylene glycol, formamide, dimethyl formamide and γ -butyrolactone;
 - about 0.05 to 30%, by weight, at least one acid selected from sulphuric acid, boric acid and phosphorus oxy acid.
- 10 2. An electrolytic capacitor comprising an anode, cathode and an electrolyte, said electrolyte further comprising:
 - about 35-60%, by weight water;
 - about 10-55%, by weight organic solvent selected from a group consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene glycol; polyethylene
 - 15 glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol;
 - about 0.05 to 30%, by weight, at least one acid selected from sulphuric acid, boric acid and phosphorus oxy acid.
3. An electrolytic capacitor according to any of claims 1-2 comprising about 35-55%, by weight, organic solvent.
- 20 4. An electrolytic capacitor according to any of claims 1-3 comprising about 40-60%, by weight, water.
5. An electrolytic capacitor according to any of claims 1-4 wherein said anode is tantalum.
6. An electrolytic capacitor according to any of claims 1-5 wherein said cathode is a
- 25 conductive metal provided with a semiconductive or metal-like conductive coating.
7. An electrolytic capacitor according to claim 6 wherein said cathode is at least one element chosen from a group consisting of an oxide, a nitride, a carbide or a metal or an activated carbon.
8. An electrolytic capacitor according to claim 7 wherein said cathode comprises a
- 30 metal selected from a group consisting of tantalum, titanium, iridium, platinum, palladium, gold, silver, molybdenum, ruthenium, tungsten, zirconium, hafnium, rhodium, vanadium, osmium and niobium.

9. An electrolytic capacitor according to claim 8 wherein when said acid comprises sulphuric acid said cathode is a metal selected from a group consisting of tantalum, iridium, platinum, palladium, gold, silver, ruthenium, tungsten, rhodium, vanadium and osmium.
- 5 10. An electrolytic capacitor according to any of claims 1-9 wherein said cathode comprises a porous ruthenium oxide film provided on a tantalum substrate or a titanium substrate or an alloy thereof.
11. An electrolyte for activating a capacitor comprising:
about 35-60%, by weight water;
10 about 10-55%, by weight organic solvent with the proviso that said organic solvent excludes ethylene glycol, formamide, dimethyl formamide and γ -butyrolactone;
about 0.05 to 30%, by weight, at least one acid selected from sulphuric, boric acid and phosphorus oxy acid.
12. An electrolyte according to any of claims 1-11 wherein said acid comprises
15 sulphuric acid and at least one acid selected from boric acid and phosphorus oxy acid.
13. An electrolyte according to claim 12 wherein said acid comprises sulphuric acid, boric acid and phosphorus oxy acid.
14. An electrolyte according to claim 13 wherein said electrolytic solution comprises:
20 about 0.05 to 10%, by weight, sulphuric acid;
about 0.05 to 10%, by weight, boric acid; and
about 0.05 to 10%, by weight, phosphorus oxy acid.
15. An electrolyte according to claim 14 wherein said solvent is selected from a group consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene
25 glycol; polyethylene glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol.
16. An electrolyte according to any of claims 1-15 wherein said solvent is glycerol.
17. An electrolyte according to any of claims 1-15 wherein said solvent is 1,3-propane diol.
- 30 18. An electrolyte according to any of claims 1-15 wherein said solvent is 2-methyl-1,3-propane diol.
19. An electrolyte according to any of claims 1-15 wherein said solvent is propylene

glycol.

20. An electrolyte according to any of claims 1-15 wherein said solvent is polyethylene glycol monomethyl ether.

21. An electrolyte according to any of claims 1-20 wherein said phosphorus oxy acid
5 is orthophosphoric acid.

22. An electrolyte according to any of claims 1-20 wherein said phosphorus oxy acid is phosphorous acid.

23. An electrolyte according to any of claims 1-22 wherein said electrolyte has a pH of less than about 7.

10 24. An electrolyte according to claim 23 wherein said electrolyte has a pH of less than about 5.

25. An electrolyte according to claim 24 wherein said electrolyte has a pH of less than about 4.

26. An electrolyte according to any of claims 1-25 wherein said electrolyte has a freezing
15 point below about -30°C.

27. A capacitor comprising the electrolyte according to any of claims 1-26.

28. A method for providing a capacitor comprising the steps of:

providing a tantalum anode;

providing a cathode;

20 activating said anode and said cathode with an electrolyte comprising:

about 35-60%, by weight water;

about 10-55%, by weight organic solvent with the proviso that said organic solvent excludes ethylene glycol, formamide, dimethyl formamide and γ -butyrolactone;

25 about 0.05 to 30%, by weight, at least one acid selected from sulphuric, boric acid and phosphorus oxy acid;

wherein said electrolyte has a pH below about 5 and a freezing point below about -30°C.

29. A method according to claim 28 wherein said acid comprises sulphuric acid and at least one acid selected from boric acid and phosphorus oxy acid.

30 30. A method according to claim 29 wherein said acid comprises sulphuric acid, boric acid and phosphorus oxy acid.

31. A method according to claim 30 wherein said electrolytic solution comprises:

about 0.05 to 10%, by weight, sulphuric acid;
about 0.05 to 10%, by weight, boric acid; and
about 0.05 to 10%, by weight, phosphorus oxy acid.

32. A method according to claim 28 wherein said solvent is selected from a group
5 consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene glycol;
polyethylene glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol.

33. A method according to claim 32 wherein said solvent is glycerol.

34. A method according to claim 32 wherein said solvent is 1,3-propane diol.

35. A method according to claim 32 wherein said solvent is 2-methyl-1,3-propane
10 diol.

36. A method according to claim 32 wherein said solvent is propylene glycol.

37. A method according to claim 32 wherein said solvent is polyethylene glycol
monomethyl ether.

38. A method according to claim 28 wherein said phosphorus oxy acid is
15 orthophosphoric acid.

39. A method according to claim 28 wherein said phosphorus oxy acid is
phosphorous acid.

40. A method according to claim 32 wherein said cathode is a conductive metal
provided with a semiconductive or metal-like conductive coating.

20 41. A method according to claim 40 wherein said cathode is at least one element
chosen from a group consisting of an oxide, a nitride, a carbide of a metal or an
activated carbon.

42. A method according to claim 41 wherein said cathode comprises a metal selected
from a group consisting of tantalum, titanium, nickel, iridium, platinum, palladium,
25 gold, silver, cobalt, molybdenum, ruthenium, manganese, tungsten, iron, zirconium,
hafnium, rhodium, vanadium, osmium and niobium.

43. A method according to claim 42 wherein when said acid comprises sulphuric
acid, sufficient buffering material is added to raise the pH to a level sufficient to avoid
dissolution of components of manufacture of said capacitor.

30 44. A method according to claim 40 wherein said cathode comprises a porous
ruthenium oxide film provided on a titanium substrate.

45. A method according to claim 28 wherein said electrolyte has a pH of less than

about 4.

46. A capacitor prepared by the method according to any of claims 28-46.

47. An electrolytic capacitor comprising an anode, cathode and an electrolyte comprising:

- 5 about 35-60%, by weight water;
 about 10-55%, by weight organic solvent with the proviso that said organic solvent excludes ethylene glycol, formamide, dimethyl formamide and γ -butyrolactone;
 about 0.05 to 30%, by weight, a mixture of ammonium acetate, boric acid, phosphorus oxy acid and acetic acid.

10 48. An electrolytic capacitor according to claim 47 wherein said electrolytic solution comprises:

- about 0.05 to 10%, by weight, ammonium acetate;
 about 0.05 to 10%, by weight, boric acid;
 about 0.05 to 10%, by weight, phosphorus oxy acid.

15 49. An electrolytic capacitor comprising an anode, cathode and an electrolyte comprising:

- about 35-60%, by weight water;
 about 10-55%, by weight organic solvent wherein said solvent is selected from a group consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene glycol; polyethylene glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol;
20 about 0.05 to 30%, by weight, a mixture of ammonium acetate, boric acid, phosphorus oxy acid and acetic acid.

25 50. An electrolytic capacitor comprising an anode, cathode and an electrolyte comprising:

- about 35-60%, by weight water;
 about 10-55%, by weight organic solvent selected from a group consisting of glycerol, 1,3-propane diol; 2-methyl-1,3-propane diol; propylene glycol; polyethylene glycol monomethyl ether; N-alkyl-2-pyrrolidone and diethylene glycol;
30 about 0.05 to 30%, by weight, a mixture of ammonium acetate and acetic acid.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
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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)

IPC 7 H01G H01M C25D

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 practical, search terms used)

EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 1 313 115 A (WILSON GREATBATCH TECHNOLOGIES) 21 May 2003 (2003-05-21)	1,11,47,49,50
X	paragraphs '0018! - '0023!	1,2,4-13,15,18-29,32-37,40-46,50
A	the whole document	14,30,31,38,39,47-49
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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INTERNATIONAL SEARCH REPORT

International Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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X	paragraphs '0006!, '0011!, '0012!; claims 1-4	1-13,15, 16,19, 23-29, 32,33, 36,40-46
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A	----- US 6 480 371 B1 (MELODY BRIAN JOHN ET AL) 12 November 2002 (2002-11-12) column 5, lines 45-67 column 6, lines 20-35,55-64; claim 1 -----	1-50

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