

(12) PATENT
(19) AUSTRALIAN PATENT OFFICE

(11) Application No. AU 199642138 B2
(10) Patent No. 710119

(54) Title
Process for purifying water

(51)⁶ International Patent Classification(s)
C01F 007/48 C01F 007/74

(21) Application No: **199642138** (22) Application Date: **1996 .01 .23**

(30) Priority Data

(31) Number (32) Date (33) Country
9500264 1995 .01 .24 SE

(43) Publication Date : **1996 .08 .01**

(43) Publication Journal Date : **1996 .08 .01**

(44) Accepted Journal Date : **1999 .09 .16**

(71) Applicant(s)
Kemira Kemi Aktiebolag

(72) Inventor(s)
Magnus Kvant; Roger Barstrom

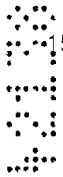
(74) Agent/Attorney
E F WELLINGTON and CO, 312 St Kilda Road, MELBOURNE VIC 3004

(56) Related Art
DER ABS ACC#92-208730/26 (CN 1054757 A)
EP 250626
CN 1086195

ABSTRACT

The present invention relates to a process for the preparation of solutions of aluminum salts, characterized in that an aluminate of an alkali metal and/or alkaline earth metal is dissolved in an acid solution for the formation of a solution
5 which is used as such or is diluted to a suitable concentration.

10

15


20




25

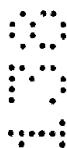

30

AUSTRALIA

PATENTS ACT 1990

REGULATION 3.2

1011-AU



Name of Applicant: KEMIRA KEMI AKTIEBOLAG



Actual Inventor/s: MAGNUS KVANT and ROGER BARSTROM



Address for Service: E.F. WELLINGTON & CO.,
Patent and Trade Mark Attorneys,
312 St. Kilda Road,
Melbourne, 3004, Victoria.



Invention Title:

"PROCESS FOR PURIFYING WATER"

Details of Associated Provisional Applications Nos:

The following statement is a full description of this invention including the best method of performing it known to us.



DESCRIPTION

Technical field

The present invention relates to a process for purifying water.

- 5 The object of the present invention is to obtain a rational and cost efficient process for purifying water, involving the use of certain aluminates and inorganic acid materials.

Background of the invention

10
15
20
25
30
35
40
45
50
55
60
65
70
75
80
85
90
95
100
105
110
115
120
125
130
135
140
145
150
155
160
165
170
175
180
185
190
195
200
205
210
215
220
225
230
235
240
245
250
255
260
265
270
275
280
285
290
295
300
305
310
315
320
325
330
335
340
345
350
355
360
365
370
375
380
385
390
395
400
405
410
415
420
425
430
435
440
445
450
455
460
465
470
475
480
485
490
495
500
505
510
515
520
525
530
535
540
545
550
555
560
565
570
575
580
585
590
595
600
605
610
615
620
625
630
635
640
645
650
655
660
665
670
675
680
685
690
695
700
705
710
715
720
725
730
735
740
745
750
755
760
765
770
775
780
785
790
795
800
805
810
815
820
825
830
835
840
845
850
855
860
865
870
875
880
885
890
895
900
905
910
915
920
925
930
935
940
945
950
955
960
965
970
975
980
985
990
995

In purification of water, different types of aqueous solutions of metal salts, such as aluminum sulphate, aluminum chloride, ferri chloride, and the similar are used. The basic polysalts of aluminum chloride and aluminum sulphate or mixed aluminum-ferri chloride are in this case particularly effective and of interest. Common to all processes for the manufacture of such solutions are that they all require high temperatures and concentrated acids in order to have the dissolution and reaction with the minerals, which are the source of the metal salts, to run with a reasonable speed. Due to these process parameters all equipment has to be made of corrosion resistant materials, as well as the equipment materials shall all be able to resist high pressures in those cases an elevated pressure is used to speed up the process.

Further the process capacity of the plants is limited due to the fact that the reaction time is long, two hours being the shortest time, and is hereby due for the more simple products, such as aluminum sulphate and aluminum chloride. More advanced products, such as the basic salt solutions require a longer time and e.g., polyaluminum chloride-sulphate requires 6 hrs at 160°C to be produced.

- 25 There is thus a well grounded demand for obtaining better processes which can increase and cost-reduce the production of such aluminum and ferri salt solutions, in particular since the world community today requires increased water purification of economically weak nations and communities as well.



Description of the present invention

In accordance with the invention, there is provided a process for purifying water comprising the steps of forming a solution from an aluminate and an acid in the absence of any oxidant, wherein the aluminate is selected from the group consisting of calcium aluminate and magnesium aluminate and the acid is selected from the group consisting of an inorganic acid, including residual acids, and aluminum chloride, whereby a solution of a basic polyaluminium salt is formed; and subsequently treating impure water with the solution.

As starting material for the present process, solid alkali aluminates, such as portland cement like minerals of the type CIMENT FONDU, calcium-aluminum-iron-titanium-silicate cement, can be used. These cement types having high aluminum contents are commercially available and the production thereof is well known. See for example The Chemistry of Cement and Concrete by F.M. Lea, E. Arnold (Publishers) Ltd. 3rd ed. pp 490-527. These cements contain typically 30 to 75 % of Al_2O_3 . They are produced by heating aluminum containing minerals, such as bauxite and aluminum hydroxide with calcium carbonate, whereby carbon dioxide is released and the cement is formed. This is then ground to a fine powder for further use. These types of cement are usually used for applications which shall resist high temperatures.

It has now turned out that these cements dissolve immediately in diluted acids, such as 10-15 % hydrochloric acid in an extremely short time, and that the compounds obtained have the same properties as those prepared in a conventional way. Thus a cement fondu comprising 40 % Al_2O_3 dissolves in 15 % hydrochloric acid within 5 minutes without any addition of heat and at atmospheric pressure, whereby a mixed polyaluminum chloride, a so called PAX, having 75 % basicity is obtained. Furthermore, the product showed improved properties in flocculating tests, which properties were superior to the properties of conventionally produced products of polyaluminum chloride having the same basicity.

Solid aluminates, such as cement fondu or sintered aluminates can be produced using different starting alkali metals or alkaline earth metals, such as calcium, magnesium, and sodium. The aluminates can be produced by sintering or melting a mixture of aluminum hydroxide and suitable salts of calcium, magnesium and



sodium. Hereby the oxides or carbonates are preferably used. Other methods for the production of alkali aluminates are known, as well.

5 The present invention is not restricted to the use of ciment fondu, but other solid aluminates produced in a different way can be used in the same way.

Example 1

A calcium aluminate cement obtained from LaFarge Fondu International, 157, Av. Charles de Gaulle, F-92521 Neuilly-sur-Seine, France was used for dissolution.

10 The cement had a typical composition as follows:

Al ₂ O ₃	50 %
CaO	40 %
Fe ₃ O ₄	17 %

15 25 grammes of this cement was mixed with 100 g of 16 % hydrochloric acid having a temperature of 10°C. After about 5 min. the reaction had ceased. The solution now contained a polyaluminum-ferri chloride having

4.95 % Al
1.7 % Fe
20 Basicity 65 %.

Example 2

25 For the preparation of a calcium aluminate, aluminum hydroxide was mixed with calcium oxide or calcium carbonate in a molar ratio of 0.30 to 2.0:1 of calcium:aluminum, whereupon sintering was carried out at 1000 to 1400°C for 0.5 to 2.5 hrs. Sintering can be carried out at a temperature of 700 to 1900°C, whereby the time for completing the sintering is changed correspondingly.

30 Dissolution of sintered aluminate
10 g of calcium aluminate having a mol ratio Ca:Al of 1.0:1 were mixed with 37 g of 37 % hydrochloric acid whereby a complete dissolution was obtained within 5 min. The dissolution provided a polyaluminum solution having an Al content of 5.5 % and a molar ratio OH/Al = 1.5, i.e. a basicity of 50%.

35 10 g of calcium aluminate having the molar ratio Ca:Al 0.5:1 were mixed with 38 g

of 37 % hydrochloric acid, which provided a polyaluminum solution having an Al content of 6.3 and a molar ratio OH/Al = 1.19, i.e. a basicity of 40%.

Example 3

- 5 Calcium and magnesium aluminates, respectively can be produced in different ways. Thereby one can add aluminum hydroxide and a salt of calcium or magnesium to sodium hydroxide, whereupon the mixture is boiled for 1 to 6 hrs. The aluminate formed precipitates and is filtered off. Alternatively, a calcium salt or magnesium salt can be added to a hot or cold sodium aluminate solution whereby
- 10 an aluminate of calcium or magnesium precipitates which can be filtered off. In both cases there is an excess of aluminum present in the solution.

Dissolution of aluminate produced according to a wet method

- 10 g of calcium aluminate having a molar ratio Ca:Al = 1.5 were dissolved in 25 g of
- 15 37 % hydrochloric acid which provided a polyaluminum solution having an aluminum content of 4.4 % and a molar ratio OH/Al = 1.6, i.e. a basicity of 53%.

- For the dissolution of solid aluminates different inorganic acids can be used, such as HCl, HNO₃, H₂SO₄, in order to provide different coagulants, i.e. flocculating
- 20 agents for the purification of e.g. sewage water. But also an aluminum chloride solution, which is acidic, can be used for the preparation of the aluminum solutions from the aluminate, whereby it is obtained that the aluminum content and the basicity can be further increased. The efficiency of a coagulant is dependent of the basicity of the solution. The desire to reach high contents of aluminum is mostly
- 25 dependent on transport economical reasons.

- By means of the present invention acids having lower concentrations than usual can be used, which is of importance as such acids are considerably more safer to handle, produce considerably smaller impact on the environment at the dissolution
- 30 and furthermore they have a much lower price. Thus residual acids from the pickling of metal objects can be used. Hereby such acids very often contains iron and aluminum which means that these residual products can be reclaimed. Today such acids are commercially completely unprofitable and be obtained for a small cost as no use for these acids is at hand and as they are a waste problem for the
- 35 industry.

For the precipitation of impurities in the purification of water, solutions having a concentration of 1 to 2 % of Al are used.

SECRET

SECRET

SECRET



The claims defining the invention are as follows:

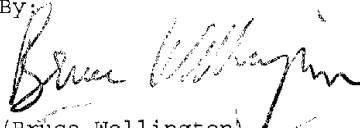
- 5 1. A process for purifying water comprising the steps of forming a solution from an aluminate and an acid in the absence of any oxidant, wherein the aluminate is selected from the group consisting of calcium aluminate and magnesium aluminate and the acid is selected from the group consisting of an inorganic acid, including residual acids, and aluminum chloride, whereby a solution of a basic polyaluminium salt is formed; and subsequently treating 10 impure water with the solution.
2. The process of claim 1 wherein the aluminate is comprised in a cement.
3. The process of claim 1 wherein the aluminate has a ratio of alkali/Al greater than 0.3.
- 15 4. The process of claim 1 wherein the aluminate is prepared according to a wet process.
5. The process of claim 1 wherein the acid is an inorganic acid.
- 20 6. The process of claim 1 wherein the acid is a residual acid.
7. The process of claim 1 wherein the acid is at least 10% HCl.
8. The process of claim 1 wherein the acid is an acidic solution of aluminum salt.
- 25 9. The process of claim 1 wherein the solution is formed without the addition of heat.
10. The process of claim 1 wherein the solution is formed at atmospheric pressure.

11. The process of claim 1, substantially as hereinbefore described with reference to any one of the practical Examples.

5 12. Purified water when obtained by the process of any one of claims 1 to 11.

DATED this 3rd day of March 1999

KEMIRA KEMI AKTIEBOLAG,
By its Patent Attorneys,
E. F. WELLINGTON & CO.,
By


(Bruce Wellington)

0
0
0
0
0

0
0
0
0

0
0

