COMMONWEALTH OF AUSTRALIA

Patents Act 1952

610150

APPLICATION FOR A STANDARD PATENT OR A STANDARD PATENT OF ADDITION

Form 1
Regulation 9

ACCEPTED AND AMENDMENTS 21-2-91

ALLOWED

KWE, ISCOR LIMITED and SCIENTIFIC CONSTRUCTION CC
of ROGER DYASON ROAD, PRETORIA, TRANSVAAL, REPUBLIC OF SOUTH AFRICA and 134 MOUNT STREET, BRYANSTON, SANDTON, TRANSVAAL, REPUBLIC OF SOUTH AFRICA respectively.

hereby apply for the grant of a ~~standard patent~~ ~~of addition~~ for an invention entitled "PROCESS FOR THE TREATMENT OF CONTAMINATED EMULSION."

which is described in the accompanying ~~provisional~~ complete specification.

(To be included in the case of a Convention application)

Details of basic application(s) –

Number of basic application 87/27006

Name of Convention country in which basic application was filed SOUTH AFRICA

Date of basic application APRIL 15, 1987

(To be included in the case of an application made by virtue of section 51)

Number of original application

Person by whom made

(To be included in the case of an application for a patent of addition)

I request that the patent may be granted as a patent of addition to the patent applied for on Application No. Patent No.

in the name of

I request that the term of the patent of addition be the same as that for the main invention or so much of the patent for the main invention as is unexpired.

My address for service is KELVIN LORD AND COMPANY, PATENT AND TRADE MARK ATTORNEYS 4 DOURO PLACE, WEST PERTH, WESTERN AUSTRALIA, AUSTRALIA 6005

Dated this 13th day of APRIL 1988

To :

THE COMMISSIONER OF PATENTS

(Signature)

ISCOR LIMITED and SCIENTIFIC CONSTRUCTION CC
By their Patent Attorneys
KELVIN LORD AND COMPANY

TRADE MARKS SUB-OFFICE 14APR1988 PERTH, W.A.

This form must be accompanied by either a provisional specification (Form 9 and true copy) or by a complete specification (Form 10 and true copy).

* These sections are to be completed only where applicable.

C.J. Thompson, Commonwealth Government Printer

THE PATENTS ACT 1952

DECLARATION IN SUPPORT OF AN APPLICATION FOR A PATENT

In support of the Application made for a patent for an invention entitled:

ert
le of Invention

ert
ll Name(s) and
dress(es)

I/We ABRAHAM CAREL GREYLING AND ALAN WAINWRIGHT LAKE
of ROGER DYASON ROAD, PRETORIA, TRANSVAAL, REPUBLIC OF SOUTH AFRICA, 134 MOUNT STREET, BRYANSTON, SANDTON, TRANSVAAL, REPUBLIC OF SOUTH AFRICA
do solemnly and sincerely declare as follows:—

sert
ll Name(s) of
pplicant(s)

1. ~~I am/We are the applicant(s) for the patent~~

(or, in the case of an application by a body corporate)

1. I am/We are authorised by
ISCOR LIMITED AND SCIENTIFIC CONSTRUCTION CC RESPECTIVELY
the applicant(s) for the patent to make this declaration on its/their behalf.

2. ~~I am/We are the actual inventor(s) of the invention referred to in the basic application(s)~~

(or, where a person other than the inventor is the applicant)

ull Name(s) and
ddress(es) of
nventor(s)

2. ALAN WAINWRIGHT LAKE
134 MOUNT STREET, BRYANSTON, SANDTON, TRANSVAAL,
REPUBLIC OF SOUTH AFRICA

ate how Applicant(s)
rive title from inventor(s)
The Applicant(s)
are the assignee(s) of the
vention from the
v.ntor(s)

is/are the actual inventor(s) of the invention and the facts upon which the applicant(s) is/are entitled to make the application are as follows:–

THE APPLICANTS ARE THE ASSIGNEES OF THE INVENTOR BY VIRTUE OF ASSIGNMENT DATED 8TH APRIL 1988

ote: Paragraphs
and 4 need only be
mpleted for a
onvention Application

asic Country(ies)
iority Date(s)
asic Applicant(s)

3. The basic application(s) as defined by Section 141 of the Act was/were made

in REPUBLIC OF SOUTH AFRICA ..... on 15 APRIL 1987
by ALAN WAINWRIGHT LAKE .....
in ..... on .....
by .....

4. The basic application(s) referred to in paragraph 3 of this Declaration was/were the first application(s) made in a Convention country in respect of the invention(s) the subject of the application.

Declared at PRETORIA this 11TH day of APRIL 19 88

A C. GREYLING
GROEPSEKRETARIS
GROUP SECRETARY
Vanor Bpk./Iscor Ltd.
.....
Signature of Declarant(s)

A.W. LAKE - SCIENTIFIC CONSTRUCTION CC
MEMBER

To: The Commissioner of Patents
KELVIN LORD AND CO.
~~G.R. CULLEN & COMPANY~~

(12) PATENT ABRIDGMENT (11) Document No. AU-B-14620/88
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 610150

(54) Title
PROCESS FOR THE TREATMENT OF CONTAMINATED EMULSION

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(57) Claim

1. A process for the treatment of a contaminated emulsion, comprising a spent metal rolling process lubricant contaminated with iron fines, the emulsion comprising an internal phase which is water, and an external phase which is a fat, in order to recover the external phase substantially free of iron fines, including the steps of:

(a) if the water content of the untreated emulsion relative to that of the rest of the emulsion is less than 50% on a mass to mass basis, mixing the contaminated emulsion with a volume of water sufficient to increase the total water content to at least 50% on the said basis;

(b) mixing the emulsion or the product of step (a) with a sufficient amount of a short-chain organic acid to lower the pH thereof to less than 5;

(c) if such components are not already present in a sufficient amount in the emulsion, mixing the product of

.../2

step (b) with an amount of a suitable emulsifier and, if required, an amount of a suitable demulsifier so that the total amount of emulsifier and/or demulsifier is sufficient to achieve a suitable separation of the external phase from the internal phase; and

(d) allowing the products of steps (b) or (c) to settle to form an upper layer comprising the external phase substantially free of water and contaminants; a lower aqueous layer; and a precipitate containing the contaminants.

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COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Application Number: Class Int. Class

Lodged:

Complete Specification Lodged:

Accepted:

Published:

This document contains the amendments made under Section 49 and is correct for printing.

Priority:

Related Art:

Name of Applicant(s): ISCOR LIMITED and SCIENTIFIC CONSTRUCTION CC

Address of Applicant(s): ROGER DYASON ROAD, PRETORIA, TRANSVAAL, REPUBLIC OF SOUTH AFRICA and 134 MOUNT STREET, BYRANSTON, SANDTON, TRANSVAAL, REPUBLIC OF SOUTH AFRICA respectively.

Actual Inventor(s): ALAN WAINWRIGHT LAKE

Address for Service: Kelvin Lord & Co.,
4 Douro Place,
WEST PERTH,
Western Australia 6005.

Complete Specification for the invention entitled:

PROCESS FOR THE TREATMENT OF COMTAMINATED EMULSION

P000137 27/06/88

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

THIS invention relates to a process for the treatment of a contaminated emulsion, comprising an internal or dispersed phase which is water, and an external or continuous phase, which may be an oil and/or fat, in order to recover the external phase substantially free of contaminants.

In the rest of this specification the term "fat" will be used to denote fats in both the solid and liquid form.

One example of such an emulsion is found in the hot and cold rolling processes in the metal working industry where blends of fats, for example glyceryl esters of higher fatty acids, fatty acids and mineral oils, are blended with suitable emulsifiers or dispersants to form lubricants which are used to lubricate and cool the rolling equipment. Generally, such a lubricant is applied in admixture with water and sprayed onto the working zone. The waste lubricant which washes from the working zone is collected in a suitable settling tank, and the

waste skimmed off. Usually this waste is then collected, drained, and dumped. The process of lubrication usually demands that an excess of lubricant is used, much of which accordingly ends up as a waste.

The largest use of such lubricants is with equipment used in the working of steel. In this instance the waste usually comprises a water-in-fat emulsion which includes contaminants such as iron fines, breakdown products of fats, partially hydrolysed glycerides, and iron soaps. Such a waste is difficult to treat to release the recoverable fats and, to date, many processes have been suggested for the recovery of the fats for possible re-use.

One such a known process, the so called Titzel process, involves cooking the waste under pressure at high temperature to produce a wet fatty substance containing a high proportion of unwanted iron soaps. Another process, again, comprises cooking the waste with a strong

mineral acid for a long period to break down and dissolve out all the iron. The resulting product is hard to wash in order to remove the acid, and the product also tends to emulsify with water. In neither of these processes is a fat produced which is satisfactory for direct re-use.

Although it is also known to use the two aforesaid processes successively in order to obtain an iron free end product, such a combined process leads to the generation of a large volume of iron salts in water, which is strongly acid. Thus, the use of the combined processes successively is expensive, wasteful in heating, while a residue is yielded which presents a serious effluent disposal problem.

A third process which has been suggested, is to treat the waste with a demulsifier in acidic conditions. However, this still leaves the problem of final dewatering of the recovered fat.

There is accordingly a need for an improved

process for the treatment of an emulsion such as a waste lubricant in order to recover the external phase for re-use, and it is an object of this invention to provide such a process.

According to the invention a process for the treatment of a contaminated emulsion, comprising a spent metal rolling process lubricant contaminated with iron fines, the emulsion comprising an internal phase which is water, and an external phase which is a fat, in order to recover the external phase substantially free of iron fines, including the steps of:

(a) if the water content of the untreated emulsion relative to that of the rest of the emulsion is less than 50% on a mass to mass basis, mixing the contaminated emulsion with a volume of water sufficient to increase the total water content to at least 50% on the said basis;

(b) mixing the emulsion or the product of step (a) with a sufficient amount of a short-chain organic acid to lower the pH thereof to less than 5;

(c) if such components are not already present in a sufficient amount in the emulsion, mixing the product of step (b) with an amount of a suitable emulsifier and, if required, an amount of a suitable demulsifier so that the total amount of emulsifier and/or demulsifier is sufficient to achieve a suitable separation of the external phase from the internal phase; and

(d) allowing the products of steps (b) or (c) to settle to form an upper layer comprising the external phase substantially free of water and contaminants; a lower aqueous layer; and a precipitate containing the

contaminants.

The emulsion is preferably a water-in-fat emulsion and the process of the invention is preferably designed for the recovery of the external phase of an emulsion containing contaminants, e.g. for the recovery of a fat from a water-in-fat emulsion containing contaminants. The initial product to be treated may, for example, be a used lubricant, e.g. a lubricant used in hot and/or cold rolling

processes, a margarine, a cooking fat, a mayonnaise or the like. The end product of the process of the invention is an external phase, i.e. a fat, substantially free of water and contaminants, which is suitable for re-use.

The emulsifier may, for example, comprise a partially hydrolysed ester of a poly hydric alcohol.

When the emulsion being treated is a water-in-fat emulsion, the emulsifier can be the degradation products of the fat, viz. diglycerides and monoglycerides. For other emulsions, the emulsifier may for example, be a sorbitan ester or a glycol ester.

The first step of the process of the invention is, if the water content of the emulsion is less than 50% on a mass per mass basis relative to the rest of the emulsion, to add to the emulsion an amount of water sufficient to ensure that the total volume of the water in the mixture is substantially at least equal to the volume of

the rest of the emulsion.

The second step of the process of the invention is to add to the emulsion, or to the mixture of the emulsion and water, a short-chain organic acid in an amount sufficient to lower the pH of the mixture to less than 5, preferably in the order of between 3 and 4. Preferably a short-chain organic acid is used which is both water soluble and fat-soluble.

Examples of suitable short-chain organic acids which may be used include formic acid, acetic acid, propionic acid, butyric acid, hydroxyacetic acid, benzoic acid, capric acid, caproic acid and caprylic acid. Certain of these acids have an unpleasant odour and the preferred acid to use is benzoic acid.

The third step of the process of the invention is, if these products are not already present in the emulsion in a sufficient amount, to add to the mixture resulting from the second step, an amount of a suitable emulsifier and

demulsifier. Generally, the addition of emulsifier and demulsifier will be necessary in order to achieve a suitable separation of the external phase from the aqueous component. It will be appreciated that in some instances the emulsion may already contain a sufficient amount of a suitable emulsifier or demulsifier which occur naturally in the emulsion, and that it will then not be necessary to add any additional emulsifier or demulsifier.

The emulsifier may be any suitable one such as one having a water and fat solubility at temperatures up to 70°C. It may be anionic or nonionic with an Hydrophylic-Lipophylic-Balance (HLB) in the order of between 8 and 12. Examples of suitable emulsifiers are certain phosphate esters, such as those sold under the trade name GAFAC RE610 (supplied by General Anilin Fabriek), and Hoechst-Hostaphat M.D. (supplied by Hoechst).

The demulsifier may also be any suitable one. Generally, the demulsifier will be cationic or

nonionic, soluble in fat, and with an HLB in the order of between 2 to 5. Examples of suitable demulisifiers include a fatty imidazoline such as that sold under the trade name CASAMINE R (supplied by Shell SA (Pty) Limited), and UNAMIDET (supplied by Lonza), which is a hydroxyethyl fatty acid-imidazoline.

The emulsifier may be added in an amount of 0,5 to 1 percent by weight of the total weight of the mixture of emulsion and water.

If necessary, the mixture resulting from step (c) above may be heated to a suitable temperature, preferably up to 100°C, to ensure that the external phase is in liquid form.

The fourth step of the process of the invention is to allow the mixture to settle into an upper and a lower layer, the upper layer comprising the external phase substantially free of water and contaminants, and the lower layer comprising a mildly acidic aqueous layer which is easy to dispose of with a minimal treatment. The

precipitate below the bottom layer comprises solid contaminants, such as metal fines.

The product to be treated, i.e. the contaminated emulsion, may also contain other ingredients such as synthetic esters and mineral oils.

Examples of the process of the invention will now be given.

EXAMPLE 1

A fatty lubricant containing approximately 10 percent of fatty acid was used as a lubricant and the waste collected. The waste consisted of 52 percent fat, 40 percent water and 8 percent iron fines. The waste was mixed with an equal volume of water so that the total water content was more than 50% on a mass to mass basis relative to the volume of the rest of the emulsion. This was done in order to make up for the expected water losses which would occur through evaporation during the subsequent heating process. An amount of benzoic acid was then added in a sufficient quantity to bring the

pH of the mixture to 3,5, whereafter there was added to the mixture 0,1 percent by weight of a mixture of ethoxylated imidazoline and 0,4 percent by weight of a mixture of a mono and diacid ester of phosphoric acid and a fatty alcohol. The mixture was stirred at 60°C for three hours and allowed to settle for four hours. This resulted in the formation of an upper layer of fat containing 0,5 percent free water and 0,2 percent iron fines with the percentage fatty acid being 12,5 percent; an aqueous layer containing 1 percent of emulsified fat; and a precipitate of metal fines. The fat layer was separated, centrifuged, and after the original dispersion system was added, was suitable for re-use as a lubricating oil.

EXAMPLE 2

A used tallow-based tin plate rolling oil was collected from a rolling mill as a viscous mixture of fat, water and iron fines.

A quantity of 4 metric tons of the oil at a temperature of 80°C was poured into a 12 000

litre tank fitted with a steam coil and an air sponge pipe for agitation. Approximately 6 000 litres of water were added together with 25kg of each of benzoic acid and a phosphate ester. The amount of water used was again an excess for the same reason as stated above. The mixture was heated to 80°C and agitated with air. After about 6 hours, it was noted that the viscosity of the mixture had fallen to close that of hot fat, and a sample showed water separation taking place as free droplets.

The agitation and heating were terminated and the contents were allowed to settle for about 12 hours. After this period, the oil at the top of the tank assumed a normal brown colour, with a small amount of iron fines being suspended therein. The bulk of the iron fines was contained in a precipitate in the bottom of the tank.

The oil was pumped off into drums until the water layer was reached midway down the tank. The drums were then centrifuged using an

Alfa-Laval-type forced ejection centrifuge at a speed of 6000 rpm. A clear brown oil was produced, the water and metal fines being rejected as a heavy sludge. The oil showed less particles in suspension than new oil, no iron soaps, only 0,1 percent of moisture, and it had an acid value of 32 mg KOH/g, which was partly due to the presence of residual additives which are not harmful to re-use.

The oil, mixed on a 50:50 basis with new oil, was found suitable for re-use for rolling. The calculated yield showed that close to 80% of the waste oil content was recovered. Of interest was the fact that due to the treatment under acid conditions no iron soaps were found in the recovered oil, even though present in the waste.

The iron containing precipitate and sludge were of interest as a source of iron fines having a particulate size of from 100 microns down to sub-micron. These iron fines were totally unoxidised and relatively easy to recover.

The above procedure was repeated on a laboratory scale without the addition of the aforesaid additives. The result was a dark, very viscous mass, containing fat, iron soaps, free iron and entrapped water in an amount of over 40%. This compound was found totally unacceptable as a rolling lubricant.

The process of the invention accordingly permits the treatment of an emulsion waste containing a contaminant such as iron fines in order to recover an external phase such as a fat suitable for use as lubricant, cheaply, efficiently and with little pollution. The recovered fat may be re-used, e.g. as a lubricant either as such or mixed with such additives as may be required. The iron fines may also be re-used in many applications, such as in the sintered metal industry, for example.

It will be appreciated that there are no doubt many variations in detail possible with a method according to the invention without departing from the spirit and/or scope of the appended claims.

The Claims defining the invention are as follows:-

1. A process for the treatment of a contaminated emulsion, comprising a spent metal rolling process lubricant contaminated with iron fines, the emulsion comprising an internal phase which is water, and an external phase which is a fat, in order to recover the external phase substantially free of iron fines, including the steps of:

(a) if the water content of the untreated emulsion relative to that of the rest of the emulsion is less than 50% on a mass to mass basis, mixing the contaminated emulsion with a volume of water sufficient to increase the total water content to at least 50% on the said basis;

(b) mixing the emulsion or the product of step (a) with a sufficient amount of a short-chain organic acid to lower the pH thereof to less than 5;

(c) if such components are not already present in a sufficient amount in the emulsion, mixing the product of step (b) with an amount of a suitable emulsifier and, if required, an amount of a suitable demulsifier so that the total amount of emulsifier and/or demulsifier is sufficient to achieve a suitable separation of the external phase from the internal phase; and

(d) allowing the products of steps (b) or (c) to settle to form an upper layer comprising the external phase substantially free of water and contaminants; a lower aqueous layer; and a precipitate containing the contaminants.

2. The process of Claim 1 wherein the emulsifier comprises a partially hydrolysed ester of a poly hydric alcohol.

3. The process of any one of the preceding claims wherein the emulsifier is a degradation product of the fat, viz diglycerides and monoglycerides.

4. The process of any one of Claims 1 or 2 wherein the emulsifier comprises a sorbitan ester or a glycol ester.

5. The process of any one of the preceding claims wherein the short-chain organic acid is added in an amount sufficient to lower the pH of the mixture to a value in the order of between 3 and 4.

6. The process of any one of the preceding claims wherein the short-chain organic acid is both water soluble and fat-soluble.

7. the process of any one of the preceding claims wherein the short-chain organic acid comprises one or more components of the group including formic acid, acetic acid, propionic acid, butyric acid, hydroxyacetic acid, benzoic acid, capric acid, caproic acid and caprylic acid.

8. The process of any one of Claims 1 to 6 wherein the short chain organic acid is benzoic acid.

9. The process of any one of the preceding claims wherein the emulsifier is one having a water and fat solubility at temperatures up to 70°C.

10. The process of any one of the preceding claims wherein the emulsifier is anionic or nonionic with an HLB in the order of between 8 and 12.

11. The process of any one of the preceding claims wherein the emulsifier is a phosphate ester, such as that sold under the trade name GAFAC RE610 (supplied by General Anilin Fabriek) and Hoechst-Hostaphat M.D. (supplied by Hoechst).

12. The process of any one of the preceding claims wherein the demulsifier is cationic or nonionic, soluble in fat, and with an HLB in the order of between 2 to 5.

13. The process of any one of the preceding claims wherein the demulsifier comprises a fatty imidazoline such as that sold under the trade name CASAMINE R (supplied by Shell SA (Pty) Limited) and UNAMIDET (supplied by Lonza), which is a hydroxyethyl fatty acid-imidazoline.

14. The process of any one of the preceding claims wherein the emulsifier is added in an amount of 0,5 to 1 percent by weight of the total weight of the mixture of emulsion and water.

15. The process of any one of the preceding claims wherein the mixture resulting from step (c) of Claim 1 is heated to a suitable temperature, preferably up to 100°C, to ensure that the external phase is in liquid form.

16. A process for the treatment of a contaminated emulsion, substantially as herein described with reference to the examples.

17. The recovered external phase of an emulsion being the product of any one of the preceding claims.

DATED FEBRUARY 12 1991

ISCOR LTD and SCIENTIFIC

CONSTRUCTION CC

By their Patent Attorneys

KELVIN LORD AND COMPANY

PERTH, WESTERN AUSTRALIA.