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(54) Title: A method for the emulsification of bitumen

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"A METHOD FOR THE EMULSIFICATION OF BITUMEN"

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- 5 The present invention is directed to a method for the emulsification of bitumen.

BACKGROUND TO THE INVENTION

10 Bitumen is a complex colloidal system that consists of a mixture of hydrocarbons and heteroatoms, including saturated aliphatic groups, naphthenic groups or cycloparaffins, aromatic rings and aliphatic groups with olefinic double bonds. Bitumen emulsions have many applications including soil stabilisation, cold mix preparation, dustbinding, surface dressing, road making and slurry sealing, to name but a few. However, the main use of bitumen is in the road making industry for
15 construction and maintenance. In road construction, bitumen products are typically applied in conjunction with a mineral aggregate. The strong adhesion that occurs between the bitumen and mineral aggregate enables the bitumen to act as a binder, with the mineral aggregate providing mechanical strength for the road.

20 Unfortunately, bitumen is difficult to work with at ambient temperatures since it is a highly viscous material under these conditions. It can, however, be transformed into a workable state by either applying heat (hot mixes), by blending with petroleum solvents (cutback mixes) or by emulsification with a surfactant in water to form a bitumen emulsion.

25 Bitumen can also be modified with suitable polymers in order to produce Polymer Modified Bitumen. The addition of polymers to bitumen improves the properties of the bitumen, such as its cohesive strength, and its resistance to a wider range of temperatures.

30 Previously, bitumen has been mixed with petroleum solvents to decrease the viscosity of the bitumen making it more workable. However, this can cause problems in terms of fire hazards and emissions.

35 An alternative approach to decrease the viscosity of bitumen, is the formation of bitumen emulsions.

Bitumen emulsions can be applied without the need for heating, although they are still prepared at high temperatures. Because bitumen emulsions do not require heating for application, they do not have the handling and environmental hazards associated with hot mixes. Bitumen emulsions also result in improved adhesion to aggregates and can be applied all year round in a range of weather conditions.

The emulsifier in a bitumen emulsion system has three general functions. These are:

- to reduce the interfacial tension between bitumen and water, allowing the emulsion to be formed (this is at the expense of the emulsion being less stable);
- to stabilise the emulsion, and when cooled the dispersion by providing long and short-range stabilising forces; and
- to assist the adhesion of the bitumen onto the mineral aggregates.

The emulsifier used can be an anionic, cationic, or non-ionic surfactants. There are two major classes of cationic surfactants based on their reactivity. The first are those that are ionised in an acidic environment and include amines, amidoamines and imidazolines, which are the most widely used emulsifiers. The second group is those that carry a permanent positive charge and cannot be neutralised by bases (e.g. quaternary ammonium salts). The cationic surfactants now used in the production of bitumen emulsions are generally based on long hydrocarbon nitrogen compounds such as alkylamines. These surfactants need to be under acidic conditions to form ammonium functional groups that result in a positive charge.

Generally, the majority of bitumen emulsions are made using a colloid mill. Production of the emulsion begins with the addition of the surfactant solution to the colloid mill, followed by the molten bitumen. The two phases are delivered into the mill by metered pumps or with manual control using flow meters. Bitumen is fed in at a temperature between 135°C and 150°C, and the temperature of the aqueous phase is adjusted to ensure that the emulsion produced does not exceed a temperature of approximately 95°C. The fluids are forced into a small gap between the rotor and the stator where they experience strong shear forces, which causes the molten bitumen to break into minute particles. Each bitumen particle is coated with the emulsifier during mixing, with some of the emulsifier remaining in the continuous phase.

Conventionally, bitumen emulsions are stored in electrically heated storage tanks at a temperature not exceeding approximately 95°C.

5 However, there are many problems with conventional blending and emulsification of bitumen. These include the stability of the resultant emulsion. Therefore, it is very important that the chemicals and additives used are tailored for the specific end use.

10 In addition high temperatures must be used when preparing the bitumen emulsion and during storage. On an industrial scale this can be very costly.

The present invention addresses these issues.

Statement of the Invention

15 According to a first aspect of the present invention, there is provided a method for the emulsification of bitumen comprising the following sequential steps:

Storing and heating water to a temperature of approximately 60°C to result in heated water;

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Adding an acid, emulsifying agent and stabilising agent to the heated water to achieve an emulsified water solution with a temperature between approximately 25°C to 30°C with a resultant pH in the range of approximately 1.6 to 1.9;

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Heating bitumen to approximately 135°C to 145°C form heated bitumen;

Dispersing the heated bitumen into the emulsified water solution in a colloid mill to form a bitumen emulsion with a temperature of approximately 90°C to 95°C;

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Cooling the bitumen emulsion to a temperature of approximately 85°C; and

Storing the cooled bitumen emulsion at a temperature of 85°C.

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This process is suitable for the production of bitumen emulsions on an industrial scale.

We have found that the temperature ranges for both the emulsified water and bitumen are crucial to manufacture good quality emulsions without any unnecessary problems with the production plant or problems with the physical or chemical properties of the resultant emulsion. For example, if the bitumen temperature goes too low, there are problems with the plant start-up and resultant emulsion temperature. If the bitumen temperature goes too high, there are problems with resultant emulsion temperature. In addition, if the water temperature goes too low, there are also problems with plant start-up and problems creating an appropriately emulsified water solution. If the water temperature goes too high, there are problems with the resultant emulsified water temperature. Thus, there is a fine balance in achieving the correct temperatures to ensure the production plant works efficiently and the resultant bitumen emulsion has the required physical and chemical characteristics for each end use.

The skilled man will be aware that graded bitumen is defined by its penetration number (pen), the harder the bitumen the lower the penetration number, the softer the bitumen the higher the penetration number. Graded bitumen can also be modified using specific additives (such as polymers/latex) to produce Modified Bitumen.

It will be understood that the bitumen used in the invention may be bitumen *per se* or modified bitumen or mixtures thereof. Thus, according to one embodiment of the invention bitumen is used. According to another embodiment of the invention, modified bitumen may be used. According to yet another embodiment of the invention, a mixture of differently graded bitumen and/or modified bitumen is used.

Ideally, the present invention is directed to a method for the emulsification of penetration grade bitumen within the range of 100pen to 220pen and/or modified bitumens.

According to one aspect of the invention the bitumen and the resultant bitumen emulsion are stored in electrically heated tanks with temperature control settings. This allows for better control than previously was obtainable.

According to another aspect of the invention, the required hot water is stored in a single tank and cold water is obtained direct from a separate cold water source, such as a well.

- 5 According to yet another aspect of the present invention, an emulsion/water heat exchanger allows for process water to be heated from the hot emulsions being produced.

- 10 It will be understood that the bitumen, water and emulsion ingredients (emulsifier, stabiliser, acid etc) are added together using pumps and flowmeters which are automated and provide a greater level of control than previously obtainable.

- 15 Mixing of the ingredients takes place in a colloid mill to ensure the bitumen is dispersed fully into micron sized droplets in water. It will be understood that two colloids mills may be used. This is advantageous as different bitumen emulsions require different colloid mill settings in order to get the required particle distribution. For example, one colloid mill could be set for producing unmodified bitumen emulsions and the other colloid mill could be set for producing modified bitumen emulsions

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According to another aspect of the invention the acid is added to the process using pump and flowmeter and pH control valve.

- 25 Another advantage of the present invention is the use of a water solution recycle tank. This allows for the collection of the initially produced water solution prior to plant start up, and once the plant has started to run, this water solution is dosed slowly back into the system during the production process. Thus, there is no waste generated.

- 30 It will be understood that where appropriate all % w/w or % w/v values are based on the weight of the resultant bitumen emulsion.

According to one embodiment of the present invention the acid is hydrochloric acid, preferably approximately 36% w/w hydrochloric acid. Other acids may be used.

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According to another embodiment of the present invention the desired pH is in the range of approximately 1.6 to approximately 1.9. The pH is controlled by various

control valves. We have found that the process will not work optimally above a pH of 2. A pH greater than 2 results in an emulsified water solution which is not homogenous and results in production problems down the line. Furthermore, an acidic pH lower than 1.6 is not recommended as the resultant bitumen emulsion will be too acidic for end use applications.

According to another embodiment of the present invention the emulsifier is a soap detergent.

Suitable soap detergents include but are not limited to

Emulsifier Redicote N39L® – Akzo Nobel Chemicals

Emulsifier Redicote EM44® – Akzo Nobel Chemicals

Emulsifier L60® – Ceca Chemicals

Ideally a cationic emulsifier is used. Such cationic emulsifiers are generally based on long hydrocarbon nitrogen compounds, such as alkyl amines and include the soap detergents listed above.

It will be understood that the selection of the emulsifier used in the preparation of bitumen emulsions is a critical factor in road making applications.

Optionally, kerosene or paraffin may be added as an emulsification aid during the emulsification process. Kerosene may be added from 0 to 20 % w/v. Alternatively, other fuels similar to kerosene may be used.

According to another embodiment of the present invention, the stabiliser or stabilising agent is a liquid calcium chloride solution. It will be understood that other stabilising agents may be used.

Ideally, the resultant bitumen emulsion comprises:

- From approximately 40 to 85% bitumen;
- From approximately 0.15 to 10% w/v emulsifying agent;
- From approximately 0 to 5% w/v stabilising agent;
- From approximately 0 to 1% w/v acid; and
- From approximately 10 to 65% w/v water.

Preferably, the resultant bitumen emulsion comprises:

- 5 From approximately 50 to 70% bitumen;
- From approximately 0.5 to 10% w/v emulsifying agent;
- From approximately 0 to 5% w/v stabilising agent;
- From approximately 0 to 1% w/v acid; and
- Water to 100% w/v.

- 10 Ideally a mixture of different penetration grade bitumen is used. According to one embodiment a mixture of 160 and 220 pen bitumen is used.

It will be understood that bitumen emulsions are generally unstable. The present invention provides rapid setting bitumen emulsions for conventional surface dressing during the summer period have been developed which are stable for approximately 10 to 14 days. The present invention also provides medium setting bitumen emulsions / modified bitumen emulsions for conventional surface dressing during the winter period have been developed which are stable for approximately 14 to 30 days. These are outlined in the Examples below.

20 The stability of the emulsion, which is the length of time it takes to set or 'break', is determined by the amount and/or type of emulsifier used.

25 This can be determined using a number of different laboratory techniques readily available to the skilled man.

Other bitumen emulsions can be prepared for other applications such as patching and tack-coating.

30 The following bitumen emulsions can be made:

- Emulsification of penetration grade bitumen (180 PEN) to produce a rapid setting bitumen emulsion for surface dressing.
- 35 - Emulsification of polymer modified bitumen to produce a rapid setting polymer modified bitumen emulsion for surface dressing.

- Emulsification of polymer modified bitumen to produce a rapid setting polymer modified bitumen emulsion for tack-coating of thin layer and / or porous asphalts.
- 5 - Emulsification of penetration grade bitumen (100 PEN) to produce an emulsion, and then the addition of natural or synthetic latex, to produce a modified bitumen emulsion.

10 It will be understood that the production process is controlled by an automatic processing system using PC/PLC system. Ideally pumping is controlled by PC controlled flowmeters. This allows for very accurate control of flow. In addition PC control ensures that once amount to be pumped is inputted, process is automatic. There is no need for operators to monitor pumping. The inputted volume linked to pumps for shutdown of process once completed.

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DETAILED DESCRIPTION OF THE INVENTION

The invention will be more clearly understood by the following description of some embodiments thereof, given by way of example only.

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The bitumen used in the present invention is stored in one or more steam heated storage tanks. It will be understood that the bitumen can be of any penetration grade, e.g. from 100pen to 220pen, or it may be modified bitumen.

25 It will be understood that the entire process is completely automated and is carried out using a PC/PLC. Once the recipe of the emulsion to be produced is selected the plant starts automatically.

30 The process for the preparation of the bitumen emulsion commences by pumping water, from for example a groundwater well, into a cold water storage tank.

The water in the cold water storage tank is heated by steam heating approx. 5 Tonnes of cold water to approximately 60°C.

35 The production process involves the following general steps:

Firstly, the bitumen begins to circulate through the pump and flowmeter back to the same tank, until the required production temperature of approximately 135°C to approximately 145°C is reached.

5 The colloid mill starts up.

The water solution system starts up and circulates back to a recycle tank. It is heated to approximately 60°C. Emulsifying agent, stabilizing agent and acid are added to the water to form an emulsified water solution. This process will continue until the correct water solution temperature and pH has been achieved. This is controlled by temperature control valves and pH control valves. Ideally, a temperature between approximately 25°C to 30°C with a resultant pH in the range of approximately 1.6 to 1.9 is required.

15 Kerosene may be stored in a storage vessel, and may be optionally added to the bitumen during bitumen emulsion production depending on the product to be produced. The addition of a small amount of kerosene to the bitumen can make it easier to emulsify, however, this is an optional step only.

20 Once all required parameters are reached, the emulsified water solution (with emulsifying agent, acid and stabilizing agent) is automatically directed through the colloid mill.

25 Immediately afterwards, the previously heated bitumen is directed through the mill to form the bitumen emulsion ideally with a temperature of from approximately 90°C to 95°C.

The resultant bitumen emulsion produced is then passed through a heat exchanger, which cools the bitumen emulsion to approx. 85°C.

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The bitumen emulsion is then pumped into electrically heated storage tanks and stored at approx. 85°C.

The resultant heated water is used as process water.

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Examples

Various different types of bitumen emulsions may be made according to the invention. Such bitumen emulsions differ in their precise components. These are outlined below. In particular, the amount of emulsifying agent, water, kerosene used is important.

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Materials

Emulsifying agents: Emulsifier Redicote N39L® – Akzo Nobel Chemicals
 Emulsifier Redicote EM44® – Akzo Nobel Chemicals
 Emulsifier L60® – Ceca Chemicals

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Stabilizing agent : A liquid calcium chloride solution

Example 1

15 Rapid setting emulsion

160/220 pen bitumen at approximately 69% w/v
 water at approximately 30% w/v
 kerosene at approximately 1%
 emulsifying agent at approximately 0.18% w/v
 stabilizing agent at approximately 0.04 % w/v
 approximately 0 to 1% w/v hydrochloric acid

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Example 2

25 Medium setting emulsion

160/220 pen bitumen at approximately 67% w/v
 water at approximately 31% w/v
 kerosene at approximately 2%
 emulsifying agent at approximately 0.25% w/v
 stabilizing agent at approximately 0.04 % w/v
 approximately 0 to 1% w/v hydrochloric acid

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Example 3

35 Slow setting emulsion

160/220 pen bitumen at approximately 63% w/v

- water at approximately 35% w/v
- kerosene at approximately 2%
- emulsifying agent at approximately 0.30% w/v
- stabilizing agent at approximately 0.04 % w/v
- 5 approximately 0 to 1% w/v hydrochloric acid

The invention is not limited to the embodiments hereinbefore described which may be varied in both construction and detail

CLAIMS

1. A method for the emulsification of bitumen comprising the following sequential steps:

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Storing and heating water to a temperature of approximately 60°C to result in heated water;

10

Adding an acid, emulsifying agent and stabilising agent to the heated water to achieve an emulsified water solution with a temperature between approximately 25°C to 30°C and a pH in the range of approximately 1.6 to 1.9;

15

Heating bitumen to approximately 135°C to 145°C form heated bitumen;

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Dispersing the heated bitumen into the emulsified water solution in a colloid mill to form a bitumen emulsion with a temperature of approximately 90°C to 95°C;

Cooling the bitumen emulsion to a temperature of approximately 85°C; and

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Storing the cooled bitumen emulsion at a temperature of 85°C.

2. The method according to claim 1 wherein bitumen within the range of 100pen to 220pen is used.
3. The method according to any of the preceding claims wherein bitumen and/or modified bitumen is used.
4. The method according to any of the preceding claims wherein a mixture of different penetration grade bitumens are used.
5. The method according to any of the preceding claims wherein the acid is hydrochloric acid, preferably approximately 36% w/w hydrochloric acid.

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6. The method according to any of the preceding claims wherein the emulsifying agent is a cationic emulsifier, preferably a soap detergent.
- 5 7. The method according to any of the preceding claims wherein from approximately 0% to 20% w/v kerosene is added with the emulsifying agent.
8. The method according to any of the preceding claims wherein the stabiliser is a liquid calcium chloride solution.
- 10 9. The method according to any of the preceding claims wherein the bitumen emulsion comprises:

From approximately 40 to 85% bitumen;
From approximately 0.15 to 10% w/v emulsifying agent;
15 From approximately 0 to 5% w/v stabilising agent;
From approximately 0 to 1% w/v acid; and
From approximately 10 to 65% water.
- 20 10. The method according to claim 9 wherein the bitumen emulsion comprises from approximately 50 to 70% bitumen and from approximately 0.5 to 10% w/v emulsifying agent.
11. The method according to claim 9 or claim 10 wherein the bitumen emulsion further comprises from 0 to 20% w/v kerosene.