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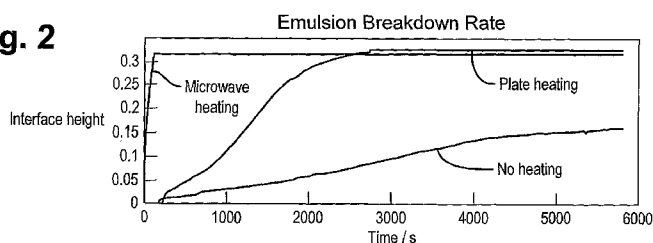
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Fig. 2



(57) Abstract: A method for separating a multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase into its component phases, the method comprising: (a) passing the multiphase fluid through an electromagnetic radiation treatment zone wherein the aqueous phase of the multiphase fluid is selectively heated by subjecting the multiphase fluid to electromagnetic radiation of frequency 1MHz to 10GHz for an exposure time of less than 5 seconds, with a power density in the aqueous phase of at least 10^7 Wm^{-3} ; (b) passing the irradiated multiphase fluid from the electromagnetic radiation treatment zone to a separator wherein the multiphase fluid is separated into its component liquid hydrocarbon and aqueous phases; and (c) withdrawing a liquid hydrocarbon stream and an aqueous stream from the separator.

A METHOD OF SEPARATING AN OIL PHASE AND AN AQUEOUS PHASE

The present invention relates to a method for separating a multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase, and in particular, for separating emulsions of produced liquid hydrocarbons and water.

In practice, produced liquid hydrocarbons, in particular, crude oils, form three layers in a separation vessel: an upper liquid hydrocarbon layer, a lower aqueous layer and an intermediate emulsion layer (often referred to as a "rag layer"). The emulsion of the intermediate emulsion layer may be stabilized by naturally occurring surfactants in the produced fluid. The demulsification of the intermediate emulsion layer can be problematic.

The use of microwave radiation to aid demulsification of water-in-oil emulsions is known. For example, US 4,582,629 describes a method for enhancing the separation of oil and water dispersions and emulsions by treating the same with microwave radiation, whether alone or in conjunction with more conventional separating devices. Thus, the emulsion or dispersion is subjected to microwave radiation in the range of one millimeter to 30 centimetres for a time sufficient to separate the dispersion or emulsion to the extent desired. Normally the microwave radiation will be used only for a short period of time, and the emulsion or dispersion will be heated to separation temperature by conventional means. The method is said to be applicable to both oil external and water external systems i.e. water-in-oil and oil-in-water emulsions respectively. The emulsion or dispersion is normally treated at a power level of from about 1 Watt to about 500 Watts per gallon (264.2 to 132,086.4 Watts per m⁻³) of emulsion or dispersion. It is said that this treatment is not sufficient to raise the temperature of the emulsion to levels normally used for separation, but will greatly enhance the resultant separation once heating is completed by conventional means. It is also said that the microwave radiation may be used in any desired wattage, but normally the higher the wattage used to produce the microwave radiation, the more quickly the dispersion or emulsion can be separated. The power used will range from a level of from about 300 to about 50,000 Watts (0.0003 to 0.05 Megawatts) and normally microwave projectors in the wattage range of from about 800 to 1000 watts will be used. For continuous treatment, a power level of from about 1 watt to about 500 watts per gallon of emulsion or dispersion will be used. The method of US

4,582,629 may be used in combination with conventional treatment vessels such as heat treaters, which are tanks modified to add heat and provide residence time in the system. Microwaves would either be used directly in the tank to generate heat and interfacial disruption, or in a section of a flow line just ahead of the tank. Likewise, the method could
5 be used in washtanks or gun barrel treaters, either in the tank or in the pipeline leading into the tank. However, it has now been found that microwave irradiation of the entire flow of multiphase fluid in a section of the flow line just ahead of the tank or microwave irradiation of the entire contents of a tank is uneconomic in terms of the power demands for the microwave generator.

10 US 4,810,375 relates to a method of enhancing the separation of oil and water emulsions and dispersions by treating the same with microwave radiation, whether alone or in conjunction with more conventional separating devices. In the method of US 4,810,375, an oil-water emulsion or dispersion is conveyed via an inlet to an applicator where it is heated by microwave energy from a power source through a circulator and wave guide.
15 The heated stream exits the applicator through an outlet to a separator vessel where the stream separates into an upper oil phase and a lower oily water phase. The separated phases are then recovered through outlets on the separator vessel. Thus, the entire feed stream to the separator vessel is subjected to microwave irradiation resulting in the separation of the feed into two layers in the separator vessel. As discussed above,
20 microwave irradiation of the entire feed stream to the separator vessel is uneconomic in terms of the power demands for the microwave generator.

WO 01/46353 describes a process comprising obtaining a used oil sample having at least 1% (by weight) aqueous substances, heating the used oil sample to a temperature of from about 20°C to about 60°C in a heating section, and extracting the heated oil by adding
25 super critical CO₂ to a demulsification section, whereby a volume of water is removed from the heated used oil sample. The heating step can be accomplished by a microwave heating process or by conventional heating techniques. A microwave generator with a power output of 6kW at 2.45GHz is applied to an emulsion flowing at 8.51 liters per minute.

30 WO 01/12289 discloses a method for treating an emulsion comprising droplets of water in an organic liquid in order to separate the water and the organic liquid, wherein the emulsion is subjected to microwave radiation with a frequency in the range from 300MHz

to 100GHz. The frequency is chosen to be within a region in which the water has an elevated tendency of transforming electromagnetic radiation into heat.

US 4853507 and US 4889639 also teach methods of treating oil/water emulsions with microwaves. US 4853507 teaches operating the system at 915MHz and 50kW input power to heat 15 gallons per minute emulsion flow to over 100°F.

Where a multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase flows through an electromagnetic radiation treatment zone, it has now been found that rapid and selective heating of the aqueous phase of the multiphase fluid may be achieved by irradiating the multiphase fluid with electromagnetic radiation having a wavelength of from 1 MHz to 10 GHz under certain critical conditions. This rapid and selective heating of the aqueous phase results in an increased rate of coalescence of emulsified droplets of aqueous phase and of macroscopic domains of aqueous phase that are dispersed in the liquid hydrocarbon phase of the multiphase fluid thereby aiding subsequent separation of the multiphase fluid into its component phases.

Thus, the present invention relates to a method for separating a multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase into its component phases, the method comprising:

(a) passing the multiphase fluid through an electromagnetic radiation treatment zone wherein the aqueous phase of the multiphase fluid is selectively heated by subjecting the multiphase fluid to electromagnetic radiation of frequency 1MHz to 10GHz for an exposure time of less than 5 seconds, with a power density in the aqueous phase of at least 10^7 Wm^{-3} ;

(b) passing the irradiated multiphase fluid from the electromagnetic radiation treatment zone to a separator wherein the multiphase fluid is separated into its component liquid hydrocarbon and aqueous phases; and

(c) withdrawing a liquid hydrocarbon stream and an aqueous stream from the separator.

Subjecting the multiphase fluid with a power density of at least $1 \times 10^7 \text{ Wm}^{-3}$ in the aqueous phase for a short period of time has been found to result in particularly fast separation times. The high power density causes selective heating of the aqueous phase which is thought to result in two mechanisms arising within the multiphase fluid. Firstly, the rapid heating of the aqueous droplets causes localized heating of the oil boundary layer around the droplets. Since oil viscosity is highly sensitive to changes in temperature, the

viscosity of the oil surrounding the droplets drops considerably, resulting in an increase in droplet settling velocity. Secondly, the rapid heating of the aqueous droplets causes the internal pressure of the droplets to increase, resulting in the expansion of the droplet. This too results in an increase in the droplet settling velocity. These mechanisms ensure that the droplets are more likely to collide and therefore more likely to coalesce with other droplets.

By the aqueous phase being "dispersed" in the liquid hydrocarbon phase is meant that a bulk layer of aqueous phase has not separated from the multiphase fluid prior to passing the multiphase fluid through the electromagnetic radiation treatment zone. The aqueous phase may be dispersed in the liquid hydrocarbon phase of the multiphase fluid in the form of emulsified droplets and/or of macroscopic aqueous domains.

Preferably, the multiphase fluid is a produced fluid from a hydrocarbon production well. The liquid hydrocarbon phase of the produced multiphase fluid may be selected from crude oils, crude oil blends, and gas condensates. The aqueous phase of the produced multiphase fluid may be selected from connate water, injection water and condensed water. Crude oils often contain naturally occurring surfactants. Accordingly, emulsified droplets of aqueous phase may arise spontaneously when a mixture of crude oil and water is subjected to shearing, for example, in a production well, choke, or a section of flow line (thereby imparting the energy required to form the emulsified droplets). Macroscopic aqueous domains may arise owing to the multiphase fluid being sufficiently well-mixed that a bulk layer of aqueous phase does not separate from the produced fluid. Thus, the flow rate of the produced fluid immediately upstream of the electromagnetic radiation treatment zone should be sufficiently high that the produced fluid does not adopt a laminar or stratified flow regime. Where the multiphase fluid is passed through a flow line prior to being passed through the electromagnetic treatment zone, the internal diameter of the flow line is preferably less than 36 inches in order to avoid the flow rate reducing to such an extent that the multiphase fluid adopts such a stratified or laminar flow regime. However, in the event that a bulk layer of aqueous phase has separated from the multiphase fluid, a mixing device may be provided upstream of the electromagnetic radiation treatment zone, for re-dispersing the bulk layer of aqueous phase in the liquid hydrocarbon phase.

Optionally, the produced fluid comprises a gaseous hydrocarbon phase, in particular, natural gas. The optional gaseous hydrocarbon phase of the produced fluid may

be entrained in the liquid hydrocarbon and aqueous phases. Alternatively, at least part of the optional gaseous hydrocarbon phase may separate into a headspace of the electromagnetic radiation treatment zone.

Where the produced fluid comprises a gaseous hydrocarbon phase, the gaseous hydrocarbon phase may be separated from the multiphase fluid in step (b). In other words, the separator employed in step (b) is a gaseous hydrocarbon-liquid hydrocarbon-water separator. Accordingly, a gaseous hydrocarbon stream is withdrawn from the separator in step (c). Alternatively, the gaseous hydrocarbon phase may be separated from the liquid hydrocarbon phase and the aqueous phase in a gas separator upstream of a liquid hydrocarbon-water separator.

It is also envisaged that the multiphase fluid may arise in a refinery operation and may therefore comprise a crude oil distillate fraction having an aqueous phase dispersed therein. Examples of crude oil distillate fractions include gasoline, gas oil, diesel, jet fuel, kerosene, heating oil and mixtures thereof.

The amount of thermal energy deposited into a material due to electromagnetic irradiation of the material is dependent upon the electric field of the electromagnetic radiation within the material to be irradiated, the frequency of the electromagnetic radiation, and the dielectric properties of the material. The liquid hydrocarbon phase and any gaseous hydrocarbon phase of the multiphase fluid are substantially transparent to the electromagnetic radiation, while the aqueous phase has a high dielectric constant and loss. The aqueous phase therefore strongly absorbs the electromagnetic radiation subsequently dissipating the resulting stored energy as heat. Accordingly, the electromagnetic radiation selectively heats the aqueous phase of the multiphase fluid thereby facilitating coalescence of the emulsified droplets of the aqueous phase and/or the macroscopic domains of aqueous phase that are dispersed in the liquid hydrocarbon phase. Typically, at least 90%, preferably, at least 95% of the electromagnetic radiation is absorbed by the dispersed aqueous phase of the multiphase fluid thereby selectively heating the aqueous phase. Suitably, the electromagnetic radiation selectively heats the dispersed aqueous phase to a temperature of at least 50°C, preferably, to a temperature in the range 60 to 100°C. Typically, the temperature differential that develops between the selectively heated aqueous phase and the liquid hydrocarbon phase of the multiphase fluid is at least 20°C, preferably, at least 50°C.

The electric field portion of the electromagnetic radiation and the exposure time of the multiphase fluid to the electromagnetic radiation are related. Accordingly, for a given multiphase fluid, the higher the electric field, the shorter the exposure time required to selectively heat the dispersed aqueous phase of the multiphase fluid. The power input

5 required to selectively heat the dispersed aqueous phase also increases with increasing amounts of dispersed aqueous phase. Thus, the power density and/or the exposure time required to selectively heat the dispersed aqueous phase increases with increasing amounts of aqueous phase dispersed in the liquid hydrocarbon phase of the multiphase fluid. Typically, the amount of aqueous phase that is dispersed in the liquid hydrocarbon phase

10 of the multiphase fluid is in the range of 0.5 to 50% by weight (based on the total weight of the aqueous phase and the liquid hydrocarbon phase).

The aqueous phase of the multiphase fluid may have salts dissolved therein. This is advantageous owing to the ionic mobility of the dissolved salts enhancing the selective heating of the dispersed aqueous phase. Preferably, the aqueous phase of the multiphase

15 fluid has a total salinity of at least 1,000 ppm, for example, at least 5,000 ppm. Examples of dissolved salts include sodium chloride.

The power density in the aqueous phase of the multiphase fluid is the energy absorbed per unit volume of the aqueous phase and can be approximated from the following Equation:

20
$$P_d = 2\pi \cdot f \cdot \epsilon_0 \cdot \epsilon'' \cdot E_0^2.$$

P_d is the power density (watts/m³)

f is the frequency of the applied energy (Hertz)

ϵ_0 is the permittivity of free space (8.854×10^{-12} F/m)

ϵ'' is the dielectric loss factor of the aqueous phase

25 E_0 is the magnitude of the electric field inside the aqueous phase (volts/m)

Suitably, the power density in the aqueous phase of the multiphase fluid in the electromagnetic radiation treatment zone is at least 1×10^7 Wm⁻³, preferably, at least 1×10^8 Wm⁻³ and optionally at least 10×10^9 Wm⁻³.

Optionally, the method may include assessing the size of droplets in the aqueous

30 phase before subjecting the multiphase fluid to electromagnetic radiation. The power density in the aqueous phase of the electromagnetic radiation can then be selected depending on the droplet size. This assessment of droplet size can be important as it has

been found that the size of the droplets in the aqueous phase has a significant effect on the speed of separation following irradiation. Figure 4 shows the separation time (t/seconds) for multiphase fluids having aqueous droplets of varying diameters (D/microns).

Increasing the power density in the aqueous phase has been found to be advantageous for smaller droplet sizes, such as droplets having a diameter less than 20microns. Droplets having a diameter of less than 20microns have a high surface area to volume ratio. This has been found to result in a high rate of heat loss to the surrounding oil phase. This rapid cooling of the aqueous phase relative to the oil phase in turn reduces the ability of the droplet to coalesce with nearby droplets. However, subjecting smaller droplets to a greater power density in the aqueous phase can ensure advantageous separation times following irradiation. This assessment can be conducted using a microscope, for example to analyse a slide dosed with a sample of the multiphase fluid, though other suitable techniques may be apparent to the skilled reader.

Typically, the multiphase fluid is exposed to the electromagnetic radiation in the electromagnetic radiation treatment zone for less than 5 seconds, preferably, less than 3 seconds, more preferably, less than 1 second. Suitably, the exposure time may be achieved by selecting a flow rate of the multiphase fluid through the electromagnetic radiation treatment zone that achieves the desired exposure time. Generally, the exposure time will be fixed by the flow rate of the multiphase fluid through an existing produced fluid flow line or through an existing refinery flow line. Accordingly, the electric field strength is varied to produce the desired treatment regime.

Without wishing to be bound by any theory it is believed that the relatively high power density of the electromagnetic radiation together with the relatively short exposure time results in rapid and selective heating of the dispersed aqueous phase in step (a) thereby enhancing separation of the aqueous and liquid hydrocarbon phases in the separator in step (b) of the present invention.

Preferably, the electromagnetic radiation is microwave radiation or radio frequency radiation. Microwave or radio frequency radiation is defined herein as electromagnetic radiation in the frequency range of 1 MHz to 10 GHz. However, in practice, the frequency of microwave radiation that is licensed for use in industrial processes is restricted to 13.56 MHz, 27.12 MHz, 40 MHz, 80 MHz, 433 MHz, 896 MHz, 915 MHz, 922 MHz and 2.45 GHz in order to avoid the risk of any accidentally emitted microwave radiation disrupting

communications equipment. Unexpectedly, given the relationship between frequency and power density set out in the above equation, it has been found that lower frequencies, particularly radio frequencies, are advantageous in the present invention. Preferably, the electromagnetic radiation has a frequency between 1MHz to 3GHz, and more preferably
5 between 1MHz to 1GHz. Frequencies in this range have been found to be more compatible for heating multiphase fluids flowing through conduits commonly used in the oil industry such as pipelines having diameters up to 36inches (91.4cm) diameter. Higher frequencies have been found to be less effective at heating the aqueous phase of a multiphase fluid in the pipelines due to their shorter wavelengths. Additionally, the
10 dielectric loss factor for the aqueous phase is higher in this frequency range.

Suitably, the desired power density is produced by developing a distribution of electric field such that the maxima in the electric field are sufficiently high to enhance the rate of separation of the multiphase fluid into its component phases. Typically, the electric field is contained within an applicator having dimensions (of size) that allow the applicator
15 to comprise part of a flow line.

The electromagnetic radiation treatment zone may be located in a flow line such that the multiphase fluid is passed from the flow line to the separator.

Suitably, radio frequency radiation may be produced using at least two electrodes that are positioned spaced apart within the electromagnetic radiation treatment zone.

20 Suitably, microwave radiation may be produced using at least one microwave generator such as a single mode or a multimode microwave cavity. Typically a plurality of microwave generators are provided. The number of microwave generators will be dependent upon the power of each generator and the total power input required for selectively heating the dispersed aqueous phase of the multiphase fluid.

25 The multiphase fluid may be subjected to microwave irradiation in the presence of an additional heating source, an electrostatic field, an acoustic field, or combinations thereof in order to further enhance coalescence of the emulsified droplets of aqueous phase and/or of the macroscopic domains of aqueous phase.

Where a part or section of flow line comprises a microwave cavity, the part or
30 section of the flow line may be provided with at least one microwave transparent window through which the microwave radiation may be transmitted into the multiphase fluid from a wave guide that is external to the section of flow line. Suitable external wave guides

would be well known to the person skilled in the art. Typically, the external wave guide is a hollow pipe wave guide which may be pressurized, for example, with dry process air. Typically, the window in the section of flow line is formed from KevlarTM, sapphire, quartz glass, or any other suitable material that is transparent to microwave radiation.

- 5 Typically, the section of flow line may be provided with a plurality of windows through which microwave radiation is transmitted from dedicated wave guides into the multiphase fluid. Preferably, the windows in the section of flow line are spaced apart in an annular pattern in order to provide a uniform field strength within the flowing multiphase fluid.

It is also envisaged that microwave radiation may be transmitted into the
10 multiphase fluid using a waveguide that penetrates through the wall of the section of flow line. Where the section of flow line is at an elevated pressure, a pressure seal is provided between the wave guide and the wall of the section of flow line, for example, a flange connector. Suitably, the wave guide that penetrates through the wall of the section of flow line, is a hollow pipe wave guide having a first end and a second end wherein the first end
15 receives microwave radiation from the microwave generator and the second end of the wave guide is provided with a window that is transparent to microwave radiation such that the microwave radiation is transmitted into the multiphase fluid where the microwave radiation is selectively absorbed by the dispersed aqueous phase (emulsified droplets of aqueous phase and/or macroscopic aqueous domains) thereby resulting in coalescence of
20 the droplets and/or domains. Generally, the hollow pipe wave guide is formed from a conductor such as silver, copper, aluminium or brass and is filled with nitrogen or another inert gas of high dielectric strength. If necessary, the hollow pipe wave guide is provided with an outer housing that is capable of withstanding the elevated pressure prevailing in the section of flow line. The window in the wave guide may be formed from, for example,
25 KevlarTM, sapphire, or quartz glass, or any other suitable material that is transparent to microwave radiation and is capable of withstanding the pressure in the section of flow line. Where the section of flow line is at an elevated pressure, a pressure seal is provided between the window and the wave guide. The second end of the wave guide may be located within the flow of multiphase fluid or where there is a headspace in the flow line,
30 the wave guide may be located in the headspace such that the microwave radiation is transmitted through the window in the wave guide and the gaseous phase that has separated into the headspace and into the multiphase fluid. Where the multiphase fluid is treated as it

flows through a section of flow line, it is envisaged that the flow line may be of a substantially circular cross-section. Typically, a plurality of microwave generators are employed, each of which has a wave guide that penetrates through the wall of the section of flow line. Preferably, the wave guides that penetrate the wall of the section of flow line are arranged spaced apart in an annular pattern around the section of flow line.

It is preferred that the multiphase fluid is treated in an electromagnetic radiation treatment zone that is arranged immediately upstream of the separator, for example, within 1 metre of the separator vessel.

Where the treated multiphase fluid is separated in step (b) using a separator vessel, the multiphase fluid separates into an upper liquid hydrocarbon layer and a lower aqueous layer. Typically, a liquid hydrocarbon stream is removed from the upper liquid hydrocarbon layer and an aqueous stream is removed from the lower aqueous layer. An intermediate emulsion layer comprising a water-in-oil emulsion may also form between the upper liquid hydrocarbon layer and lower aqueous layer. However, it has been found that the amount of intermediate emulsion layer is substantially reduced compared with operation of the separation vessel in the absence of selective heating of the aqueous phase of the multiphase fluid in an electromagnetic radiation treatment zone. Thus, the rate at which any intermediate emulsion layer builds up in the separator vessel is substantially reduced. Accordingly, it may not be necessary to heat the contents of the downstream separator to effect separation of the multiphase fluid into its component phases.

Alternatively, the heating duty for the downstream separator vessel may be substantially reduced compared with operating a separator vessel in the absence of selective heating of the aqueous phase in an electromagnetic radiation treatment zone immediately before the separator vessel. A further advantage of the present invention is that the residence time of the multiphase fluid in the separator vessel is substantially reduced compared with operation of the separator vessel in the absence of selective heating of the aqueous phase in the electromagnetic radiation treatment zone. Suitably, the residence time of the multiphase fluid in the separator vessel is less than 10 minutes, preferably, less than 5 minutes. The separator vessel is therefore capable of processing high amounts of the multiphase fluid. Typically, the separator vessel processes 50,000 to 100,000 barrels of multiphase fluid per day.

Another advantage of the process of the present invention is that the size and number of the downstream separator vessels may be reduced compared with a conventional separation of the multiphase fluid. Yet a further advantage of the process of the present invention is that it is envisaged that the water content of the hydrocarbon phase that is withdrawn from the separator vessel (or from the final separator vessel of a series of vessels) may be less than 0.5 weight% thereby allowing for a reduction in the size of an electrostatic coalescer that is used to remove low amounts of water from the separated liquid hydrocarbon stream.

As discussed above, the treated multiphase fluid may be subjected to further heating in the separator vessel to assist in separating the liquid hydrocarbon and aqueous phases. This further heating raises the temperature of the liquid hydrocarbon phase. However, an advantage of the irradiation treatment of the present invention is that the temperature to which the liquid hydrocarbon phase is heated is substantially lower than in the absence of selective heating of the aqueous phase of the multiphase fluid. For example, where it is necessary to bulk heat the multiphase fluid in a separator vessel to a temperature of 90°C to effect separation of its component liquid hydrocarbon and aqueous phases, it is envisaged that separation of the multiphase fluid in a separator vessel arranged downstream of the electromagnetic radiation treatment zone may be achieved at a temperature of as low as 60°C for the liquid hydrocarbon phase. Preferably, the irradiated multiphase fluid is heated in the separator vessel such that the liquid hydrocarbon phase reaches a temperature of from about 45 to 115°C, although temperatures of about 50 to 65°C are preferred. Typically, the pressure within the separator vessel is in the range of 5 to 50 bar.

It has been found that the power demand for the method of the present invention is significantly less than a method that relies on bulk heating of the entire multiphase fluid that flows from a flow line into a separator vessel (i.e. where there is no temperature differential between the aqueous and liquid hydrocarbon phases). Typically, the power demand is less than 50%, preferably, less than 25% of the power demand for a method that relies on bulk heating of the entire multiphase fluid in a separator.

It is envisaged that the treated multiphase fluid that is introduced into the separator vessel may be passed through an inlet device, for example, a cyclone or baffles to assist in separating any gaseous hydrocarbon phase into a head space of the separator vessel. The

inlet device may have the further advantage of reducing the turbulence of the multiphase fluid such that the multiphase fluid adopts a laminar or stratified flow regime in the separator vessel thereby assisting separation of the multiphase fluid into its component phases.

5 Although the present invention has been described in relation to separation of the multiphase fluid into its component phases in a separator vessel, it is also envisaged that the treated multiphase fluid may be separated using alternative separators, for example, using a centrifugal separator, a hydrocyclone or t-junction separator. Such separators are well known to the person skilled in the art.

10 It is envisaged that the method of the present invention may not be in continuous use. Thus, where prior art techniques for separating a multiphase fluid are satisfactory, the multiphase fluid will not be subjected to electromagnetic irradiation. The method of the present invention may then be deployed as the volume of intermediate emulsion layer begins to build up in, for example, a separator vessel. During deployment of the method of
15 the present invention, it is preferred that the multiphase fluid is either subjected to a plurality of pulses of electromagnetic radiation or is continuously subjected to electromagnetic radiation in the electromagnetic radiation treatment zone. Where the multiphase fluid is subjected to a plurality of pulses of electromagnetic radiation, the exposure time is based on the summed duration of the pulses.

20 In a further embodiment of the present invention, the multiphase fluid may be pre-treated by being passed into a separator vessel where the multiphase fluid separates into an upper liquid hydrocarbon layer, a lower aqueous layer and an intermediate water-in-oil emulsion layer. Optionally, a gaseous hydrocarbon phase may separate into a headspace of the separator vessel. Accordingly, an emulsion stream comprising a water-in-oil emulsion
25 may be withdrawn from the intermediate emulsion layer and may be subjected to electromagnetic irradiation outside of the separator vessel in accordance with the present invention. Thus, the term multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase encompasses an emulsion comprising emulsified droplets of aqueous phase dispersed in a continuous liquid hydrocarbon phase. Pre-treatment of the
30 multiphase fluid in a separator vessel is advantageous, as a portion of the aqueous phase is removed prior to subjecting the emulsion stream to electromagnetic irradiation.

Accordingly, loss of energy into any of the aqueous phase that is not in the form of emulsified aqueous droplets (i.e. into macroscopic aqueous domains) is thereby reduced.

The emulsion stream may be irradiated as it flows through a flow line which is in fluid communication with an outlet and inlet of the separator vessel. Thus, the emulsion stream may be withdrawn from the intermediate emulsion layer and may be circulated through an external recycle line where the emulsion stream is irradiated with

electromagnetic radiation in an electromagnetic radiation treatment zone before returning the irradiated (demulsified) stream to the separator vessel. Alternatively, two or more separator vessels may be arranged in series, typically, 2 or 3 separator vessels. The

emulsion stream withdrawn from the intermediate emulsion layer of the first separator vessel in the series flow through a flow line where the emulsion stream is subjected to electromagnetic radiation in an electromagnetic radiation treatment zone before passing the irradiated (demulsified) stream to the second separator vessel in the series. If an emulsion layer begins to build up in the second separator vessel in the series, an emulsion stream is

removed from the intermediate emulsion layer of the second separator vessel in the series and flows through a flow line where it is subjected to electromagnetic irradiation in an electromagnetic radiation treatment zone before passing the irradiated (demulsified) stream to the third separator vessel in the series. Preferably, a section of the flow line(s) acts as a tubular microwave chamber arranged such that microwave radiation penetrates radially

into the flowing emulsion stream. Suitably, the microwave chamber supports an appropriate distribution of electric field for treating the flowing emulsion stream. The emulsion stream may be withdrawn from the intermediate emulsion layer of the separator tank using at least one withdrawal means, for example, at least one nozzle located within the intermediate emulsion layer. The withdrawal means may be attached to and carried by a float device which is adapted to float within the intermediate emulsion layer such that the emulsion stream is withdrawn therethrough and is passed into the flow line where the emulsion stream is to be subjected to electromagnetic irradiation. Alternatively, a plurality of withdrawal means may be located at different levels within the separator vessel wherein each withdrawal means has an open and closed position. Suitably a level instrument is

provided for determining the position of the intermediate emulsion layer in the separator vessel. Accordingly, one or more of the withdrawal means that are located within the intermediate emulsion layer is opened while any of the plurality of withdrawal means

located outside the intermediate emulsion layer (for example, in the hydrocarbon layer or aqueous layer) are closed. Also, the withdrawal means may be fixed in place in the separator vessel and the position of the intermediate emulsion layer may be controlled such that the withdrawal means lies within the emulsion layer, for example, by adjusting the rate of withdrawal of the aqueous stream from the lower aqueous layer. Where the irradiated stream is recycled to the separator vessel, a pump is preferably located in the flow line upstream of the location where the emulsion is subjected to electromagnetic irradiation. Positioning the pump downstream of the location in the flow line where the emulsion is subjected to electromagnetic irradiation should be avoided as there is a risk of the emulsion reforming owing to the pump shearing the irradiated (demulsified) stream. Where the irradiated emulsion stream is recycled to the separator vessel, the withdrawn emulsion stream is suitably subjected to electromagnetic irradiation at the elevated pressure prevailing in the separator vessel thereby avoiding the need to de-pressurize the emulsion stream and to re-pressurize the irradiated (demulsified) stream. Where the irradiated stream is passed to a further separator vessel, this further separator vessel is preferably at a lower pressure than the vessel from which the emulsion stream is withdrawn such that there is no requirement for a pump.

Optionally, a demulsifier may be added to the multiphase fluid to enhance the rate of separation into its component phases. Where the multiphase fluid is treated as it flows through a flowline, the emulsifier should be added at a sufficient distance upstream of the location where the multiphase fluid is subjected to electromagnetic irradiation in order to achieve good mixing of the emulsifier with the multiphase fluid. Where an emulsion stream is withdrawn from the intermediate emulsion layer and is irradiated outside of the separator vessel, the demulsifier is preferably added to the withdrawn emulsion stream upstream of the electromagnetic radiation treatment zone and preferably, upstream of any pump. The demulsifier may be selected from any known demulsifier that is not degraded by microwave irradiation. Typically, the demulsifiers will have a molecular weight in the range 500 to 5000, preferably 500 to 2000. The demulsifier suitably has a hydrophilic-lipophilic balance value of above 9, preferably in the range 9 to 30, for example, 9 to 15. Demulsifiers which will not degrade when exposed to microwave irradiation include those which contain functional groups such as ethers, amines, ethoxylated alcohols, sulfonates and mixtures thereof.

The amount of demulsifier added to the multiphase fluid is typically in the range of 5 to 100 ppm, preferably, 10 to 25 ppm based on the amount that enters the hydrocarbon phase. Additionally, a delivery solvent may be employed. The delivery solvent is suitably miscible with the liquid hydrocarbon phase of the multiphase fluid. Where the hydrocarbon phase is a crude oil, the delivery solvent may be, for example, a crude oil distillate boiling in the range of about 70 to 450°C. Where the oil phase is a crude oil distillate fraction, the delivery solvent may, for example, be the crude oil distillate fraction or a component thereof. It is also envisaged that the delivery solvent may be a conventional solvent that is miscible with the oil, for example an alcohol, ether, or mixture thereof. The delivery solvent may be present in an amount of from 30 to 75% weight in the demulsifier.

The present invention will now be illustrated by reference to Figures 1, 2 and 3 and to the following Examples.

Example 1

An apparatus was employed that allowed an oil/water emulsion to be exposed to an electric field for a pre-determined period of time. The resultant exposure time for each batch of emulsion was approximately 0.5 seconds.

The experiments were carried out on 250ml oil/water emulsions where the oil phase was a crude oil from the Harding Field in the North Sea.

Experiments were carried out at input powers of 3, 4, 5 and 6kW, resulting in a power density in the aqueous phase of $3.5 \times 10^7 \text{ Wm}^{-3}$, $5.4 \times 10^7 \text{ Wm}^{-3}$, $6.65 \times 10^7 \text{ Wm}^{-3}$ and $8 \times 10^7 \text{ Wm}^{-3}$ respectively. It was found that an input power in excess of 7kW resulted in almost immediate emulsion separation. The results from these studies are presented in Figure 1 which illustrates separation time versus single mode microwave power for the 250ml oil/water emulsions using a pass-through microwave exposure technique.

The results show that separation time is greatly enhanced by increasing the microwave power density. Microwave treatment in single mode cavities typically produces much higher power densities (power density is proportional to electric field squared) than its equivalent multimode cavity. The results of these single mode studies suggest that a short exposure (of less than 1 second) in a high power density cavity results in a significant reduction in oil/water separation time.

Comparative Example 1

Emulsions 1 to 3 were prepared using identical mixing routines with each emulsion comprising 70% by volume Harding crude oil and 30% by volume water. Emulsion 1 was subjected to microwave irradiation for 160 seconds in a 800 W multimode microwave cavity; Emulsion 2 was treated by conventional heat transfer via a heat plate set to a temperature of 100°C; and Emulsion 3 was left untreated thereby acting as a control.

The hot plate experiment was designed to give a relative assessment of microwave heating versus conventional heating. The results of the experiments (separation rate comparison) are presented in Figure 2. It was found that microwave irradiation of Emulsion 1 resulted in a 90% reduction in separation time compared with the hot plate treatment of Emulsion 2 and a 98% reduction in separation time compared with the untreated emulsion.

Example 2

An apparatus was employed that allowed an oil/water emulsion to flow through a microwave treatment zone and into a storage vessel. Tests were carried out at 2.45GHz and at microwave powers of 3kW, 6kW, 9kW, 12kW and 15kW, resulting in a power density in the aqueous phase of $5.19 \times 10^7 \text{ Wm}^{-3}$, $1.04 \times 10^8 \text{ Wm}^{-3}$, $1.56 \times 10^8 \text{ Wm}^{-3}$, $2.08 \times 10^8 \text{ Wm}^{-3}$ and $2.60 \times 10^8 \text{ Wm}^{-3}$ respectively.

For each power density, a multiphase fluid flow rate of 6 l/min, 9 l/min and 12 l/min was tested, resulting in exposure times of approximately 2 seconds, 1.5 seconds and 1 second respectively.

The experiments were carried out on emulsions having 80% oil to 20% water, where the oil phase was a crude oil from the Harding Field in the North Sea.

Figure 3 shows the separation time (t/seconds) against microwave power (P/kW) for the 2 second (points A), 1.5 second (points B) and 1 second (points C) flow rates. It is readily seen that significantly reduced separation times are achieved with increasing power densities. Also, it can be seen that at a power density in the aqueous phase of $2.60 \times 10^8 \text{ Wm}^{-3}$, a very short exposure time (1 second) achieved an almost identical separation time as that achieved with a 1.5 second exposure time and a very similar separation time as that achieved with a 2 second exposure time. Accordingly, at this high power density, a very short exposure time gave substantially the same results as exposure times up to twice as long.

Claims

1. A method for separating a multiphase fluid comprising an aqueous phase dispersed in a liquid hydrocarbon phase into its component phases, the method comprising:
 - 5 (a) passing the multiphase fluid through an electromagnetic radiation treatment zone wherein the aqueous phase of the multiphase fluid is selectively heated by subjecting the multiphase fluid to electromagnetic radiation of frequency 1MHz to 10GHz for an exposure time of less than 5 seconds, preferably, less than 1 second, with a power density in the aqueous phase of at least $1 \times 10^7 \text{ Wm}^{-3}$, preferably, at least $1 \times 10^9 \text{ Wm}^{-3}$;
 - 10 (b) passing the irradiated multiphase fluid from the electromagnetic radiation treatment zone to a separator wherein the multiphase fluid is separated into its component liquid hydrocarbon and aqueous phases; and
 - (c) withdrawing a liquid hydrocarbon stream and an aqueous stream from the separator.
2. A method as claimed in Claim 1, wherein the multiphase fluid is subjected to
15 electromagnetic radiation of frequency 1MHz to 1GHz.
3. A method as claimed in Claim 1 or Claim 2, wherein the multiphase fluid is a produced fluid from a hydrocarbon production well.
4. A method as claimed in Claim 3 wherein the produced fluid additionally comprises a gaseous hydrocarbon phase and the multiphase fluid is separated into its component
20 gaseous hydrocarbon, liquid hydrocarbon and aqueous phases in step (b) either using a gaseous hydrocarbon-liquid hydrocarbon-water separator or a gas separator that is located upstream of a liquid hydrocarbon-water separator.
5. A method as claimed in Claim 1 or Claim 2 wherein the multiphase fluid comprises a crude oil distillate fraction having an aqueous phase dispersed therein.
- 25 6. A method as claimed in any one of the preceding claims wherein at least 90%, of the electromagnetic radiation is absorbed by the dispersed aqueous phase of the multiphase fluid thereby selectively heating the aqueous phase.
7. A method as claimed in any one of the preceding claims wherein the electromagnetic radiation selectively heats the dispersed aqueous phase to a temperature of
30 at least 50°C, preferably, to a temperature in the range 60 to 100°C.

8. A method as claimed in any one of the preceding claims wherein a temperature differential of at least 20°C, preferably, at least 50°C develops between the selectively heated aqueous phase and the liquid hydrocarbon phase of the multiphase fluid.

9. A method as claimed in any one of the preceding claims wherein the aqueous phase of the multiphase fluid has salts dissolved therein.

10. A method as claimed in any one of the preceding claims wherein the electromagnetic radiation treatment zone is a section of flow line comprising a microwave cavity and the section of the flow line is either provided with at least one microwave transparent window through which microwave radiation is transmitted into the multiphase fluid from a wave guide that is external to the section of flow line or microwave radiation is transmitted into the multiphase fluid using a waveguide that penetrates through the wall of the section of flow line.

11. A method as claimed in any one of claims 1 to 9, wherein the electromagnetic radiation treatment zone is a section of flow line in which at least two electrodes are located in spaced apart arrangement for producing radio frequency radiation.

12. A method as claimed in any one of the preceding claims wherein the multiphase fluid is passed into a separator vessel where the multiphase fluid separates into an upper liquid hydrocarbon layer, a lower aqueous layer and an intermediate water-in-oil emulsion layer, and wherein an emulsion stream comprising a water-in-oil emulsion is withdrawn from the intermediate emulsion layer and is passed through an electromagnetic radiation treatment zone where the emulsion stream is subjected to electromagnetic irradiation thereby forming an irradiated stream.

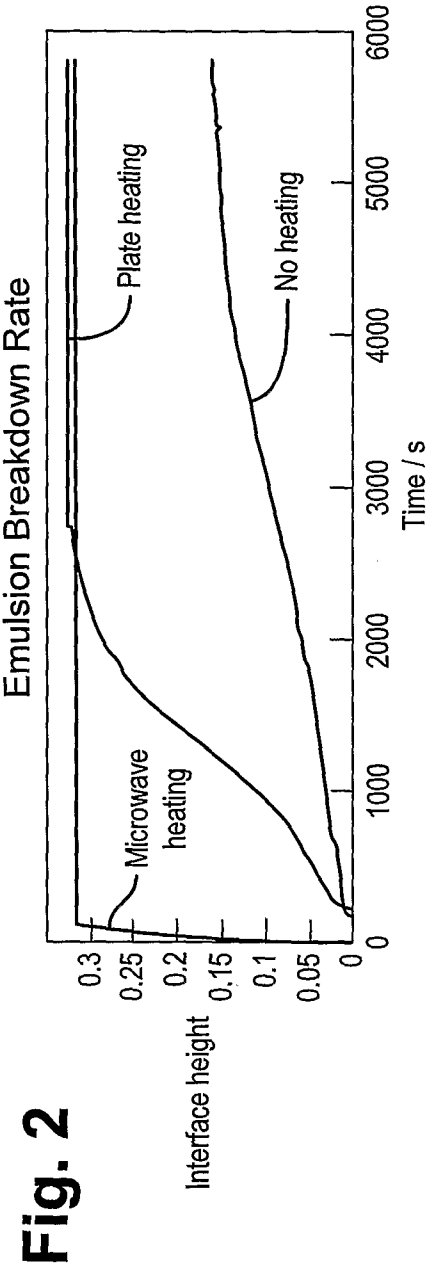
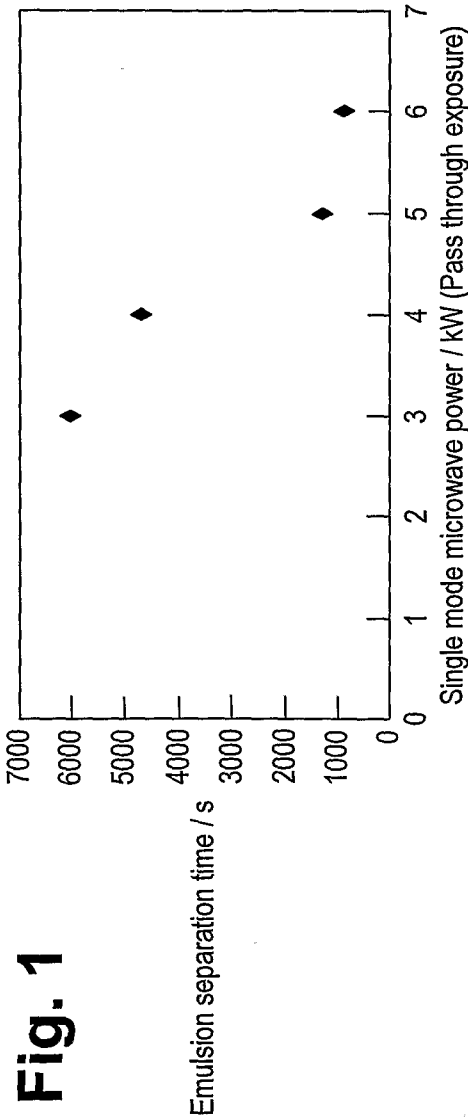
13. A method as claimed in Claim 12 wherein the emulsion stream is passed through a recycle line that is in fluid communication with an outlet and inlet of the separator vessel wherein a section of the recycle line comprises the electromagnetic radiation treatment zone and the emulsion stream is subjected to electromagnetic radiation in the electromagnetic radiation treatment zone thereby forming the irradiated stream that is recycled to the separator vessel.

14. A method as claimed in Claim 12 wherein two separator vessels are arranged in series, the emulsion stream is withdrawn from the intermediate emulsion layer of the first separator vessel in the series and flows through a flow line wherein a section of the flow line comprises an electromagnetic radiation treatment zone and the emulsion stream is

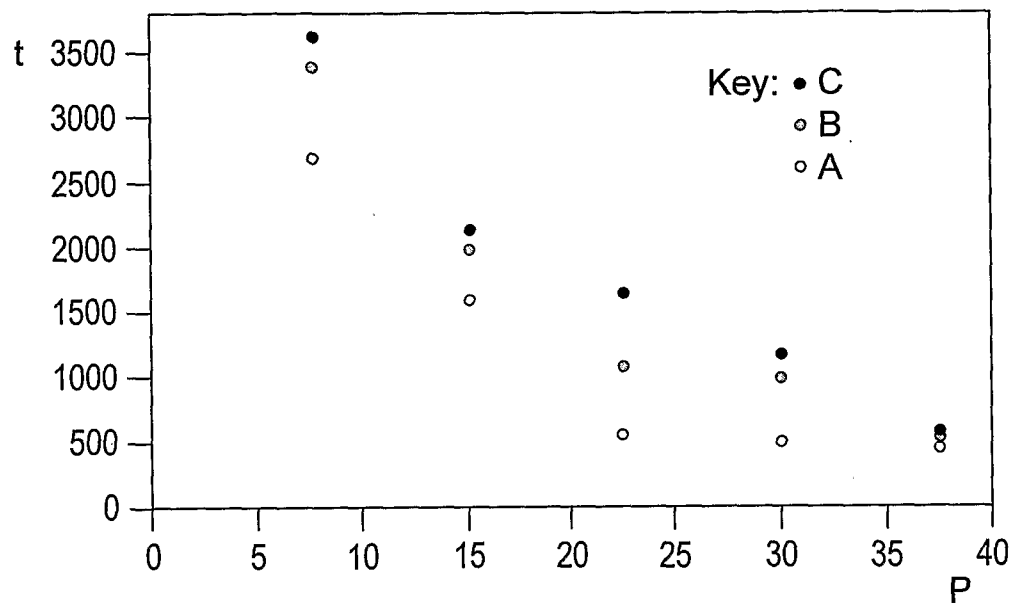
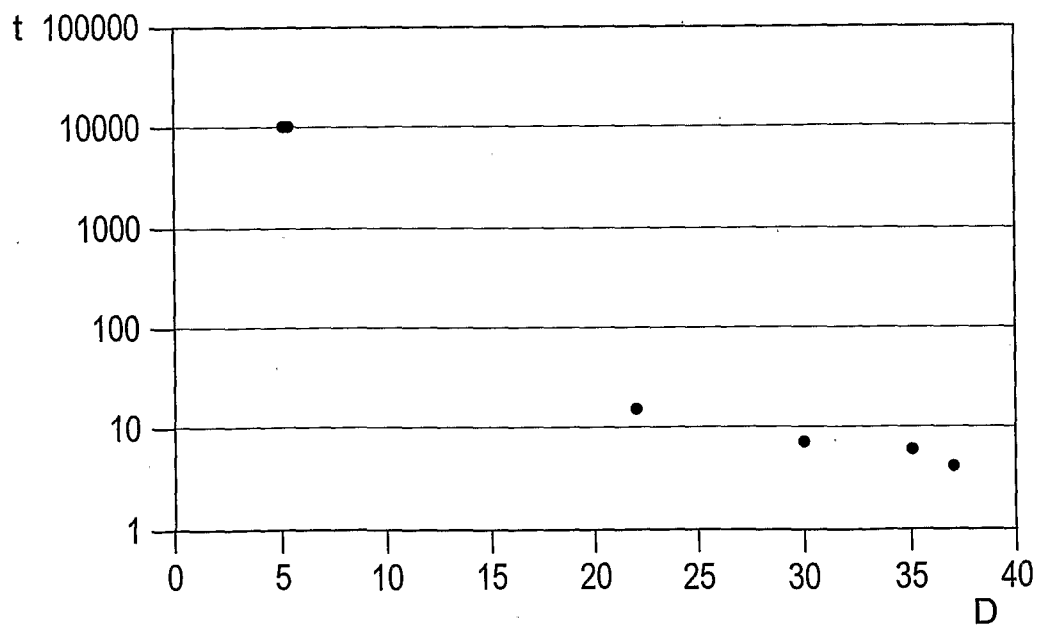
subjected to electromagnetic radiation in the electromagnetic radiation treatment zone thereby forming the irradiated stream that is passed to the second separator vessel in the series.

15. A method as claimed in any one of the preceding claims wherein a demulsifier is
5 added to the multiphase fluid to enhance the rate of separation of the multiphase fluid into its component phases.

16. A method as claimed in any one of the preceding claims, further comprising the
step of assessing the size of droplets in the aqueous phase and selecting the power density
in the aqueous phase depending on the droplet size, wherein the smaller the droplets in the
10 aqueous phase, the higher the power density.



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Fig. 3**Fig. 4**

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2008/000747

A. CLASSIFICATION OF SUBJECT MATTER INV. B01D17/04 C10G32/02 C10G33/02 C02F1/30		
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) B01D C10G C02F		
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		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 01/12289 A (ABB RESEARCH LTD [CH]; NILSEN PAAL J [NO]; KORNFELDT ANNA [SE]; NYGREN) 22 February 2001 (2001-02-22)	1-12, 14, 15
Y	page 2, line 18 - line 29 page 3, line 14 - line 24 page 6, line 13 - page 7, line 6 page 7, line 15 - line 20	16
X	WO 01/46353 A1 (IND MICROWAVE TECHNOLOGIES INC [US]; DUQUESNE UNIVERSITY [US]) 28 June 2001 (2001-06-28) page 11, line 13 - line 22 page 12, line 29 - line 39 page 13, line 14 - line 33 page 14, line 36 - page 15, line 4 claims 1,3-5; figures 4-6	1,6-12
-/--		
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : *A* document defining the general state of the art which is not considered to be of particular relevance *E* earlier document but published on or after the international filing date *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) *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 *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 *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 *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. *&* document member of the same patent family		
Date of the actual completion of the international search 6 June 2008		Date of mailing of the international search report 18/06/2008
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer Roider, Josef

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2008/000747

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 582 629 A (WOLF NICHOLAS O [US]) 15 April 1986 (1986-04-15) cited in the application column 2, line 14 - line 48 column 3, line 32 - line 40 claims 1-11 -----	1-9, 12, 15
X	US 4 853 507 A (SAMARDZIJA NIKOLA [US]) 1 August 1989 (1989-08-01) column 3, line 58 - column 4, line 9 column 5, line 58 - column 6, line 63 -----	1-12, 15
X	US 4 889 639 A (HUDGINS ROGER L [US] ET AL) 26 December 1989 (1989-12-26) cited in the application column 2, line 36 - line 68 column 3, line 23 - line 32; claim 1 -----	1-9, 12, 13
Y	HOLTZE ET AL: "The microwave absorption of emulsions containing aqueous micro- and nanodroplets: A means to optimize microwave heating" JOURNAL OF COLLOID AND INTERFACE SCIENCE, ACADEMIC PRESS, NEW YORK, NY, US, vol. 302, no. 2, 15 October 2006 (2006-10-15), pages 651-657, XP005644252 ISSN: 0021-9797 paragraph 3.Results, page 653, right-hand column, line 22 - line 28 -----	16

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2008/000747

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 0112289	A	22-02-2001	AU 6740600 A	13-03-2001
			BR 0012990 A	30-04-2002
			CN 1370089 A	18-09-2002
			NO 993968 A	19-02-2001
			OA 12152 A	05-05-2006
			ZA 200200710 A	25-04-2003
WO 0146353	A1	28-06-2001	AU 2912401 A	03-07-2001
			US 7045038 B1	16-05-2006
US 4582629	A	15-04-1986	NONE	
US 4853507	A	01-08-1989	NONE	
US 4889639	A	26-12-1989	NONE	