

**(12) STANDARD PATENT**  
**(19) AUSTRALIAN PATENT OFFICE**

(11) Application No. **AU 2016202370 B2**

(54) Title  
**Compositions and methods of making compositions**

(51) International Patent Classification(s)  
**C08J 3/00** (2006.01)

(21) Application No: **2016202370**

(22) Date of Filing: **2016.04.14**

(43) Publication Date: **2016.05.05**

(43) Publication Journal Date: **2016.05.05**

(44) Accepted Journal Date: **2018.04.26**

(62) Divisional of:  
**2009268290**

(71) Applicant(s)  
**Smith & Nephew Orthopaedics AG**

(72) Inventor(s)  
**Brunner, Lorenz;Sander, Jonathan;Dirix, Yvo**

(74) Agent / Attorney  
**Davies Collison Cave Pty Ltd, Level 15 1 Nicholson Street, MELBOURNE, VIC, 3000, AU**

## Abstract

5 A method for obtaining a composition of at least two components, comprising the steps of: providing at least one first fluid component; providing at least one second solid component and processing it so that the first component can diffuse into the second component; and diffusing the first component into the second component. A composition prepared by such a method.

2016202370 05 Apr 2018

## Compositions and method of making compositions

This is a divisional of Australian Patent Application No. 2009268290, the entire  
5 contents of which are incorporated herein by reference.

The present invention relates to a method for obtaining a composition/mixture of at  
least two components. The present invention also relates to compositions/mixtures  
made by such methods, and devices incorporating such compositions/mixtures.

10

Ultra High Molecular Weight Polyethylene (UHMWPE) is a versatile material  
combining high strength and toughness with high wear resistance. As a result, it is  
used in many industrial applications such as bearings, gearings, liners, chain guides,  
for example. A problem with processing UHMWPE results from its extremely high  
15 melt viscosity (zero shear viscosity  $> 10^8$  Pa.s), which does not allow common  
processing techniques such as injection molding or extrusion to be used. Instead,  
UHMWPE powder is sintered and then the part is mechanically machined into the  
desired shape. Since conventional melt-processing and mixing techniques are not  
applicable, blending of additives is normally done by mixing the UHMWPE powder  
20 with the additive followed by sintering. The mixing of the two powders is difficult since  
the UHMWPE powder has a very low density and is highly porous. If the additive is  
also a powder, it is difficult to form a homogeneous powder mixture. If the additive is  
in liquid form, homogeneous distribution of that liquid in the powder having an  
extremely high surface area is also difficult.

2016202370 05 Apr 2018

It is known to mix additives with the UHMWPE powder or diffuse them into the UHMWPE powder before the sintering step. It is also known to diffuse the additives into the sintered products.

- 5 According to the present invention, there is provided a method for obtaining UHMWPE containing at least one additive comprising: providing UHMWPE in the form of a powder; compacting the UHMWPE at a temperature below the melting temperature of the UHMWPE and at a pressure greater than atmospheric pressure, for a time, temperature, and pressure sufficient to form at least a first dense body
- 10 comprising deformed porous powder particles formed from UHMWPE, wherein the deformed porous powder particles of the first dense body are not completely fused and comprise narrow channels throughout the first dense body, wherein the narrow channels are capable of supporting rapid and uniform liquid adsorption into the first dense body due to capillary forces in the narrow channels; providing at least a first
- 15 additive as a liquid; and exposing at least a portion of the first dense body to the first additive as a liquid, wherein the liquid is drawn into the first dense body by capillary forces in the narrow channels of the first dense body thereby forming a composition comprising UHMWPE and the at least first additive.
- 20 The compacting pressure may be in the range from 2 to 15 MPa, preferably about 10 MPa.

The compacting temperature may be in the range of from 10 to 30°C less than the melting temperature of the UHMWPE.

05 Apr 2018  
2016202370

The compacting temperature may be about 120°C.

The first additive may comprise at least one solid dissolved in a liquid.

- 5 The method may further comprise the step of removing the liquid from the first dense body while the at least one solid of the first additive remains in the first dense body, thereby forming a composition comprising UHMWPE and the at least one solid of the first additive.
- 10 The liquid may be removed from the first dense body by heating the composition comprising UHMWPE and the at least first additive to a temperature below the melting temperature of the UHMWPE.

- The method may further comprise providing at least a second additive as a liquid; and
- 15 exposing composition comprising UHMWPE and the at least first additive to the second additive as a liquid, whereby the second additive is drawn into the first dense body by capillary forces in the narrow channels of the first dense body acting on the liquid and thereby carrying the second additive into the first dense body, thereby forming a composition comprising UHMWPE and the at least first and second
- 20 additives.

The second additive may comprise at least one solid dissolved in a liquid.

The method may further comprise removing the liquid of the second additive from the

2016202370 05 Apr 2018

first dense body while the at least one solid of the second additive remains in the first dense body, thereby forming a composition comprising UHMWPE, the at least one solid of the first additive and the at least one solid of the second additive.

- 5 The liquid of the second additive may be removed from the first dense body by heating the first dense body to a temperature below the melting temperature of the UHMWPE.

The method may further comprise the step of sintering the composition.

10

The first dense body may be sintered above the melting temperature of the polymer.

The sintering may comprise heating the composition in a mold for 15 minutes at a temperature of 220°C and a pressure of 5 MPa.

15

The method may further comprise the step of cross-linking the UHMWPE.

The cross-linking may be performed after sintering the composition.

- 20 The cross-linking may be performed by irradiation. The cross-linking may be performed using gamma or e-beam irradiation. The irradiation dose may be in the range from 1 to 25 MRad, preferably in the range from 3 to 20 MRad.

The first additive may comprise an antioxidant.

2016202370 05 Apr 2018

The first additive may comprise at least one Vitamin. The first component may comprise Vitamin E.

The first additive may comprise an antibiotic.

5

The antibiotic may be selected from the group consisting of gentamycin, vancomycin, streptomycin, penicillin and derivatives thereof.

10 The first additive may comprise a foaming agent. The foaming agent may be a solvent with a boiling temperature above the sintering temperature of the polymer.

According to the present invention, there is also provided a composition prepared by a method according to the present invention as defined herein.

15 The composition may be formed into an artefact.

The artefact may be a medical device. The medical device may be a hip or knee replacement.

20 The medical device may be sterilised.

According to some embodiments disclosed herein, the additives are diffused into the compacted body, i.e., into the intermediate state between powder and sintered object. During compaction below the melting temperature, the porous powder particles are

2016202370 05 Apr 2018

deformed into a dense body but since no melting occurs, the particles are not completely fused. Between the deformed particles, there are very narrow channels that support the rapid and uniform fluid absorption due to the capillary forces acting

5 locally. These capillary forces are not present between loose particles or in the sintered and completely fused product. After the additive has been soaked into the compacted body, a final sintering step is done to fuse the particles. Viscous or solid additives can be dissolved to enable soaking into the compacted body. For these embodiments,

2016202370 08 Mar 2017

the solvent can be evaporated before sintering or it evaporates during the subsequent sintering step.

The sintered materials can be used for medical implants such as total hip or knee  
5 replacements. These polyethylene implants containing additives for anti-oxidative purposes can also be cross-linked after the pre-compaction-soaking-sintering process using gamma or e-beam irradiation. The irradiation doses may vary from 1 to 25 Mrad or more preferably from 3 to 20 Mrad. The medical implants can also be sterilized either using gamma irradiation (2.5 - 4 Mrad) or  
10 surface sterilization methods such as ETO or gas plasma treatments.

Solid compositions may be processed from any type of polymer in powder form or from more than one type of polymer. If a polymer is available only in solid bulk or pellet form, the material may be grinded to a powder prior to compaction. The  
15 pressure may be chosen between 0 – 50 MPa, more preferably between 0 – 20 MPa and even more preferably between 5 – 15 MPa. The processing temperature is preferably set to a temperature below the melting temperature ( $T_m$ ) of the polymer. If two or more different polymers are processed the temperature is preferably set to a temperature below  $T_m$  of the polymer with the  
20 lowest  $T_m$ . More preferably, the temperature is set to  $T_m - 30\text{ }^{\circ}\text{C}$ , more preferably to  $T_m - 20\text{ }^{\circ}\text{C}$  and even more preferably to  $T_m - 10\text{ }^{\circ}\text{C}$ . The pressure may be applied first followed by heating of the mould. The heating of the mould may be applied first followed by the application of pressure. The compaction time depends on the volume of the solid composition and is preferably between 1  
25 second and 100 hours, more preferably between 1 minute and 24 hours and even more preferably between 30 minutes and 6 hours. All material in the processing mould should reach the desired compaction time. The temperature may be decreased prior to releasing the pressure. The pressure may be released prior to decreasing the temperature. The compaction procedure may be  
30 performed in normal air atmosphere, in vacuum environment or in an inert gas atmosphere such as nitrogen or argon.

2016202370 05 Apr 2018

In those embodiments of the invention comprising polyethylene, compaction of the polyethylene may be performed at a temperature above room temperature and below the melting temperature (25-130°C), at pressures ranging from 0.5-25 MPa (more preferably 1 to 15 MPa, even more preferably 2 to 10 MPa).

Compacted solid compositions are preferably processed from UHMWPE. For example, UHMWPE powder may be filled in a mould at room temperature and subsequently a pressure of about 10 MPa applied and maintained during the whole compaction procedure. Subsequently, the temperature is increased from room temperature to about 120 °C. At about 120 °C and about 10 MPa, the powder is kept for a period of time to completely heat all of the polymer powder to about 120 °C. The period of time depends on the volume of the solid composition, for example around 20 minutes for a composition with the dimensions 4 x 4 x 2 cm, and around 4 hours for a composition with the dimensions 20 x 20 x 5 cm. Subsequently, the temperature is decreased. Below a temperature of about 50 °C, the pressure can be released and the solid composition can be removed from the mould.

Embodiments of the present invention are further described herein below, by way of example, with reference to the following drawings and non-limiting examples, in which:

Figure 1 shows soaking of a compacted GUR 1020 block in a red isopropanol/fuchsin solution;

Figure 2 shows soaking of a sintered GUR 1020 block in a red isopropanol/fuchsin solution;

Figure 3 shows compacted and soaked blocks cut into two pieces (1% curcumin solution in acetone) after drying;

Figure 4 shows average weight change of the 2 standing blocks as a function of the soaking time;

2016202370 08 Mar 2017

Figure 5 shows vitamin E concentration profiles in the sintered blocks that were previously compacted and soaked in a Vitamin E-hexane solution;

Figure 6 shows examples of one or more soaking steps with different soaking directions; and

5        Figure 7 shows oxidation profiles of Vitamin E soaked and additive-free samples.

**Example 1: diffusion of dyes / colors into compacted UHMWPE bodies**

10        GUR 1020 UHMWPE powder was compacted in a press at 120°C and a pressure of 10 MPa. A small block (4 cm x 3cm x 5 cm) was cut from the plate and put into a glass containing 75 ml of isopropanol and 0.04 grams of Fuchsin (Merck). In Figure 1, the soaking behavior at room temperature of the pre-compacted block is depicted as a function of time. Within seconds, the fluid  
15        including the color additive is absorbed and within an hour the body is uniformly colored.

Figure 1 shows soaking of a compacted GUR 1020 block in a red isopropanol/fuchsin solution (left: seconds after immersion; middle: 30 minutes  
20        after immersion; right: 1 hr after immersion).

**Comparative example 1:**

25        A sintered block of GUR 1020 (4x3x5 cm) was put into a glass containing 75 ml of isopropanol and 0.04 grams of Fuchsin (Merck). Figure 2 shows the soaking behavior at room temperature of the block depicted as a function of time (left: seconds after immersion; middle: 30 minutes after immersion; right: 1 hr after immersion).

30        In the comparative example, the sintered block is not impregnated with the fluid.

2016202370 08 Mar 2017

**Example 2: soaking of natural additives/antioxidants into small compacted blocks**

5 GUR 1020 blocks were compacted below the melting temperature at 120°C in a laboratory scale press for 15 minutes at 10 MPa. Afterwards, the compacted blocks were rapidly cooled to room temperature.

10 Soaking: 3.8 x 4 x 1.5 cm compacted blocks were soaked at room temperature in a 1% w/w solution of acetone containing curcumin as an additive. After soaking for an hour, the acetone was evaporated in a vacuum oven at 40°C for 24 hr. The compacted and soaked block was cut into two pieces (Figure 3) showing the homogeneous distribution of the yellow curcumin

15 Figure 3 shows compacted and soaked blocks cut into two pieces (1% curcumin solution in acetone) after drying. Figure 3 (a) and (b) represent two different blocks, both cut into 2 pieces

**Example 3: soaking of antioxidants - Vitamin E into small compacted blocks followed by sintering**

20

The compaction was done as described in Example 2. After compaction the samples were immersed in a hexane-vitamin E solution (2.8% w/w) and the weight was measured during soaking. 2 compacted blocks were standing in the solution (only lower part of block immersed, see also Figure 1) and 1 block was  
25 completely covered with the soaking solution (inside the liquid).

After soaking, the samples were dried to constant weight in a vacuum oven (see example 2) and the weight was measured again to determine the VitE content in the material. Finally, the compacted polyethylene blocks were sintered for 15  
30 minutes in a mold at a temperature of 220°C and a pressure of 5 MPa. The samples were finally cooled rapidly (in 8 minutes) to room temperature.

2016202370 08 Mar 2017

FTIR measurements were conducted to determine the content of vitamin E in the samples. From the sintered blocks, small portions were cut in regular distances. From these smaller pieces, microtome slices were produced with a thickness of about 300 microns (or 5 times 60 microns). Of these slices, FTIR spectra were recorded with a Bruker Vertex 70 with a resolution of 4 cm<sup>-1</sup> and a total of 16 scans.

For a more precise determination of the vitamin E concentration, the measured spectra were normalized and a spectrum of pure UHMWPE was deducted. The 2020 cm<sup>-1</sup> peak was chosen as reference peak and its height (relative to the height at 2100 cm<sup>-1</sup> and 1980 cm<sup>-1</sup>) was normalized to an absorbance of 0.05. This is supposed to correspond to a film thickness of 100 microns. Of this normalized spectrum, the spectrum of pure UHMWPE, normalized by the same procedure, was deducted. Then, the height of the C-OH absorption (vitamin E peak) at 1210 cm<sup>-1</sup> (relative to the height at 1188 cm<sup>-1</sup> and 1231 cm<sup>-1</sup>) was determined. The concentration of vitamin E (mol/kg) was calculated according to the following equation:

$$A = \varepsilon \cdot b \cdot C$$

A = peak absorbance (height of the 1210 cm<sup>-1</sup> peak)  
 $\varepsilon$ : molar absorptivity of the  $\alpha$ -tocopherol -OH in UHMWPE (in kg·cm<sup>-1</sup>·mol<sup>-1</sup>). Experimentally determined = 133 kg·cm<sup>-1</sup>·mol<sup>-1</sup>  
 b = path length (film thickness) in cm = 0.01 cm for normalized spectra  
 C = concentration of  $\alpha$ -tocopherol in UHMWPE in mol·kg<sup>-1</sup>

In Figure 4, the average weight change of the 2 standing blocks is depicted as a function of the soaking time. Initially there is a fast weight increase within 4 hrs, afterwards, the weight increase levels off. The weight increase is due to the absorption of the hexane-vitamin E solution.

In Figure 5, the concentration profiles of Vitamin E in the blocks are shown after solvent evaporation and subsequent sintering. Figure 5 shows vitamin E

concentration profiles in the sintered blocks that were previously compacted and soaked in a Vitamin E-hexane solution. 2 blocks were standing in the solution, partially immersed in the fluid, and 1 block was completely immersed in the fluid (inside).

The weight% of Vitamin E in the UHMWPE determined from the integrated FTIR spectra and from the gravimetric method are listed below.

	Gravimetric	Integrated FTIR data
Standing #1	3.1%	2.3%
Standing #2	3.1%	2.9%
Inside	0.81%	1.15%

This example shows that it is possible to impregnate the compacted body with a solution containing Vitamin E, subsequently evaporate the solvent (hexane) and finally sinter the compacted material. The amount of vitamin E in the block can be tuned by selecting different concentrations of Vitamin E in the solution or by selecting the appropriate soaking procedure.

In accordance with embodiments of the present invention, compacted blocks can be soaked in more than one soaking step. The additive in the fluid during a second or third soaking step may be different from the first soaking step. The additive can also be a chemical cross-linking agent (such as dibenzoylperoxide) or an antibiotic (such as gentamycin) or a reactive monomer (e.g. styrene or methylmethacrylate) or a foaming agent (a solvent with boiling temperature above the sintering temperature of the polyethylene). The foaming agent can have a high boiling temperature at ambient pressure, i.e. after sintering but the foaming agent may also be liquid during sintering at elevated pressures and be in

2016202370 08 Mar 2017

the gaseous phase upon release of the pressure after sintering. Also the direction of the soaking can be different as explained in Figure 6, which shows examples of one or more soaking steps with different soaking directions.

- 5 The soaking can be also restricted to a part of the compacted object therewith creating portions in the block that contain the additive and portions without the additive. In example 1, if the compacted block was removed from the soaking fluid (left picture) the sintered product would only be partially colored. This results in portions of blended and virgin material in the compacted body. Also a
- 10 compacted and soaked body with an additive can be placed in a solvent in a second step to locally extract an additive and create concentration gradients in the compacted material.

- The current invention is not restricted to UHMWPE powders but also powders
- 15 from lower molecular weight polyethylenes such as HDPE, LDPE, LLDPE. The method can also be applied to other polymers such as PMMA, polystyrene, polypropylene, PVC, polyoxymethylene (POM), PPSU, PPO, PEEK, Polyamides (PA6, PA 6.6, PA 4.6), other polyacrylates (such as poly butylacrylate), PTFE.

- 20 Advantages of the present method include the following. For additive mixing involving powders, the capillary forces acting in the compacted body are not present between loose particles and therefore it is not possible to get a rapid, uniform and efficient fluid uptake for fluid additives. For solid additives, the present method enables a more uniform distribution of the additives by first
- 25 dissolving the additive and subsequent soaking. Of course, solid additives cannot be soaked /diffused into the compacted body without the use of a carrier liquid.

- For additive diffusion into sintered objects, the particles in the sintered objects
- 30 are fused and no capillary forces are acting between the particles that enable a rapid and efficient fluid absorption and diffusion (see comparative example 1).

2016202370 08 Mar 2017

Therefore, elevated temperatures close to the melting temperature are necessary to stimulate the classical Fick diffusion into the object which is slower and less efficient. In the present invention, additives are soaked into compacted materials at room temperature within minutes/hours which is not possible when using sintered UHMWPE parts.

Example 4: Oxidation of blocks soaked with antioxidant and irradiated with gamma radiation

The oxidation resistance of a block containing antioxidants that was gamma irradiated was determined. A block that was processed according to the method described in Example 3 (soaked with vitamin E prior to sintering) was irradiated with a dose of 14 Mrad ( $\pm 10\%$ ) in normal air atmosphere. No post-irradiation thermal treatment was applied.

Cylindrical samples with a length of 40 mm and a diameter of 10 mm were drilled out of the irradiated block. Subsequently, the samples were accelerated aged according to ASTM F 2003 in an oxygen bomb at 5 atm oxygen pressure and 70 °C for 14 days. Oxidation indices of the aged components were determined by means of FTIR according to ASTM F 2102-06. The method for making measurements of the oxidation index according to this standard is as follows: thin slices of the sample are made with a microtome and tested to give a depth profile for the oxidation index. From the micro-slices taken of the sample the infrared spectrum is taken by means of FTIR with a resolution of  $4\text{cm}^{-1}$ . The oxidation index is defined as the intensity of the peaks in the region  $1680\text{-}1765\text{cm}^{-1}$ , which is associated with carbonyl peaks, divided by the intensity in a reference band which lies between  $1330$  and  $1396\text{cm}^{-1}$ .

In Figure 7, the oxidation profile of a vitamin E soaked and irradiated (gamma in air, 14 Mrad) sample is shown. The oxidation profile is an

2016202370 05 Apr 2018

average of three individual measurements. As control sample, an UHMWPE without additive, irradiated with 14 Mrad in air (without post-irradiation thermal treatment), is shown. The reduced oxidation of the material that was soaked with vitamin E is clearly demonstrated, as the maximum oxidation index of this material is below 0.02.

The reference in this specification to any prior publication (or information derived from it), or to any matter which is known, is not, and should not be taken as an acknowledgment or admission or any form of suggestion that that prior publication (or information derived from it) or known matter forms part of the common general knowledge in the field of endeavour to which this specification relates.

Throughout this specification and the claims which follow, unless the context requires otherwise, the word "comprise", and variations such as "comprises" and "comprising", will be understood to imply the inclusion of a stated integer or step or group of integers or steps but not the exclusion of any other integer or step or group of integers or steps.

2016202370 10 Apr 2017

- 16 -

1. A method for obtaining UHMWPE containing at least one additive comprising:
  - providing UHMWPE in the form of a powder;
  - compacting the UHMWPE at a temperature below the melting temperature of the UHMWPE and at a pressure greater than atmospheric pressure, for a time, temperature, and pressure sufficient to form at least a first dense body comprising deformed porous powder particles formed from UHMWPE, wherein the deformed porous powder particles of the first dense body are not completely fused and comprise narrow channels throughout the first dense body, wherein the narrow channels are capable of supporting rapid and uniform liquid adsorption into the first dense body due to capillary forces in the narrow channels;
  - providing at least a first additive as a liquid; and
  - exposing at least a portion of the first dense body to the first additive as a liquid, wherein the liquid is drawn into the first dense body by capillary forces in the narrow channels of the first dense body thereby forming a composition comprising UHMWPE and the at least first additive.
2. The method of claim 1, wherein the first additive comprises at least one solid dissolved in a liquid.
3. The method of claim 2, further comprising removing the liquid from the first dense body while the at least one solid of the first additive remains in the first dense body, thereby forming a composition comprising UHMWPE and the at least one solid of the first additive.
4. The method of claim 3, wherein the liquid is removed from the first dense body by heating the composition comprising UHMWPE and the at least first additive to a temperature below the melting temperature of the UHMWPE.
5. The method of claim 3, further comprising:
  - providing at least a second additive as a liquid; and
  - exposing composition comprising UHMWPE and the at least first additive to

2016202370 10 Apr 2017

- 17 -

the second additive as a liquid, whereby the second additive is drawn into the first dense body by capillary forces in the narrow channels of the first dense body acting on the liquid and thereby carrying the second additive into the first dense body, thereby forming a composition comprising UHMWPE and the at least first and second additives.

6. The method of claim 5, wherein the second additive comprises at least one solid dissolved in a liquid.
7. The method of claim 6, further comprising removing the liquid of the second additive from the first dense body while the at least one solid of the second additive remains in the first dense body, thereby forming a composition comprising UHMWPE, the at least one solid of the first additive and the at least one solid of the second additive.
8. The method of claim 7, wherein the liquid of the second additive is removed from the first dense body by heating the first dense body to a temperature below the melting temperature of the UHMWPE.
9. The method of any preceding claim, wherein the compacting pressure is in the range from 2 to 15 MPa.
10. The method of claim 9, wherein the compacting pressure is about 10 MPa.
11. The method of any preceding claim, wherein the compacting temperature is in the range of from 10 to 30°C less than the melting temperature of the UHMWPE.
12. The method of any preceding claim, wherein the compacting temperature is about 120 °C.
13. The method of any preceding claim, further comprising sintering the composition.

2016202370 10 Apr 2017

- 18 -

14. The method of claim 13, wherein the sintering comprises heating the composition in a mold for 15 minutes at a temperature of 220°C and a pressure of 5 MPa.
15. The method of any preceding claim, further comprising crosslinking the UHMWPE.
16. The method of claim 15 when dependent from claim 13 or 14, wherein the crosslinking is performed after sintering the composition.
17. The method of claim 15 or 16, wherein the crosslinking is performed by irradiation.
18. The method of claim 17, wherein the crosslinking is performed by gamma irradiation or by e-beam irradiation.
19. The method of claim 18, wherein the gamma or e-beam irradiation dose is in the range of from 1 to 25 MRad.
20. The method of claim 18, wherein the gamma or e-beam irradiation dose is in the range of from 3 to 20 MRad.
21. The method of any preceding claim, wherein the at least first additive is selected from the group comprising antioxidant, vitamin E, antibiotic and foaming agent.
22. The method of claim 21 when dependent from claim 5 or from any claim dependent from claim 5, wherein the first additive and the second additive are different.
23. The use of a composition obtained by the method of any preceding claim in the manufacture of a medical device.
24. The use of claim 23, wherein the medical device is a medical implant selected from the group consisting of hip and knee replacements.

2016202370 14 Apr 2016

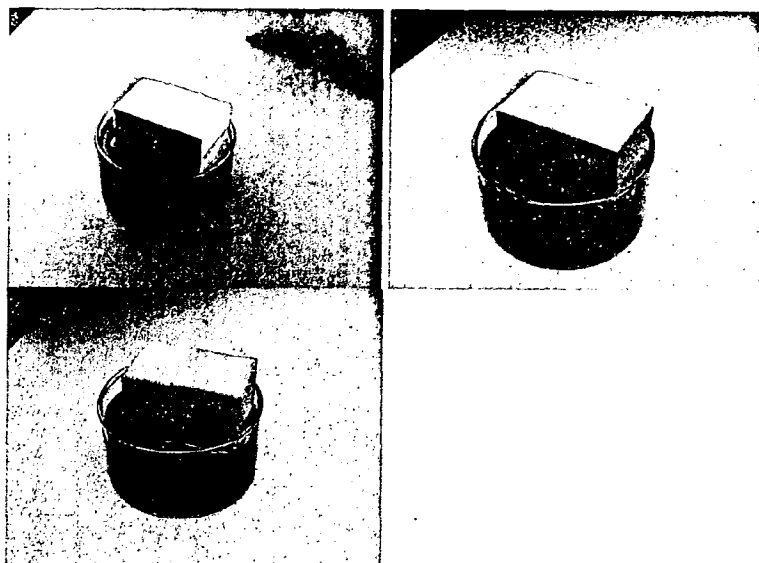
**Figures**

Figure 1: Soaking of a compacted GUR 1020 block in a red isopropanol/fuchsin solution. Left: seconds after immersion; middle 30 minutes after immersion, right 1 hr after immersion

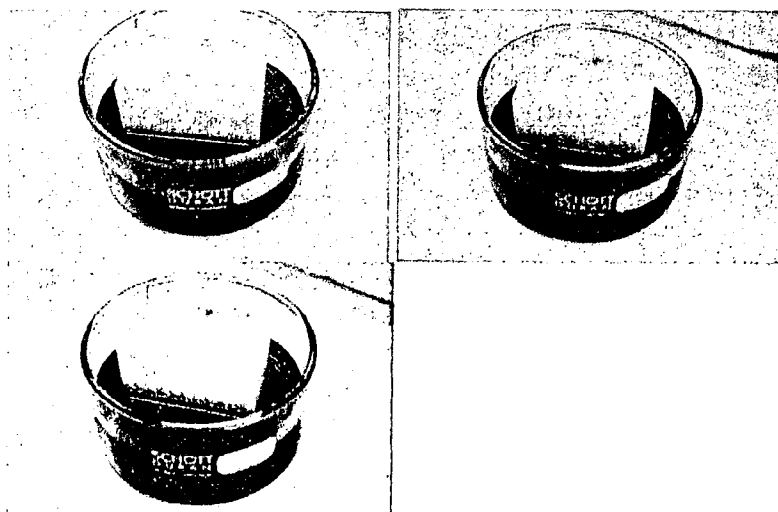


Figure 2: Soaking of a sintered GUR 1020 block in a red isopropanol/fuchsin solution. Left: seconds after immersion; middle 30 minutes after immersion, right 1 hr after immersion

a b

Figure 3: Compacted and soaked blocks cut into two pieces (1% curcumin solution in acetone) after drying. a) and b) represent two different blocks, both cut in 2 pieces

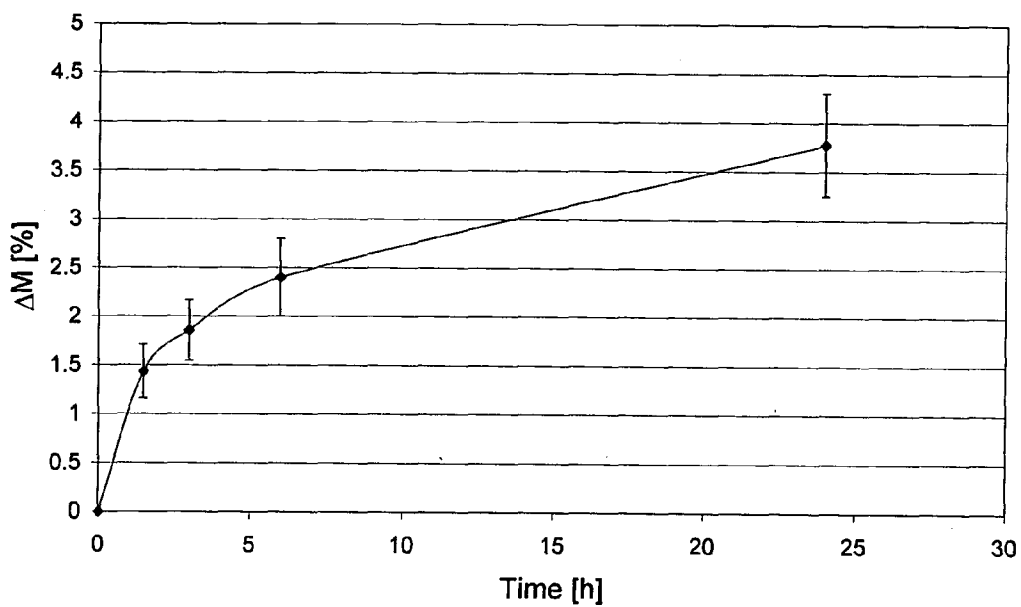


Figure 4: Average weight change of the 2 standing blocks as a function of the soaking time

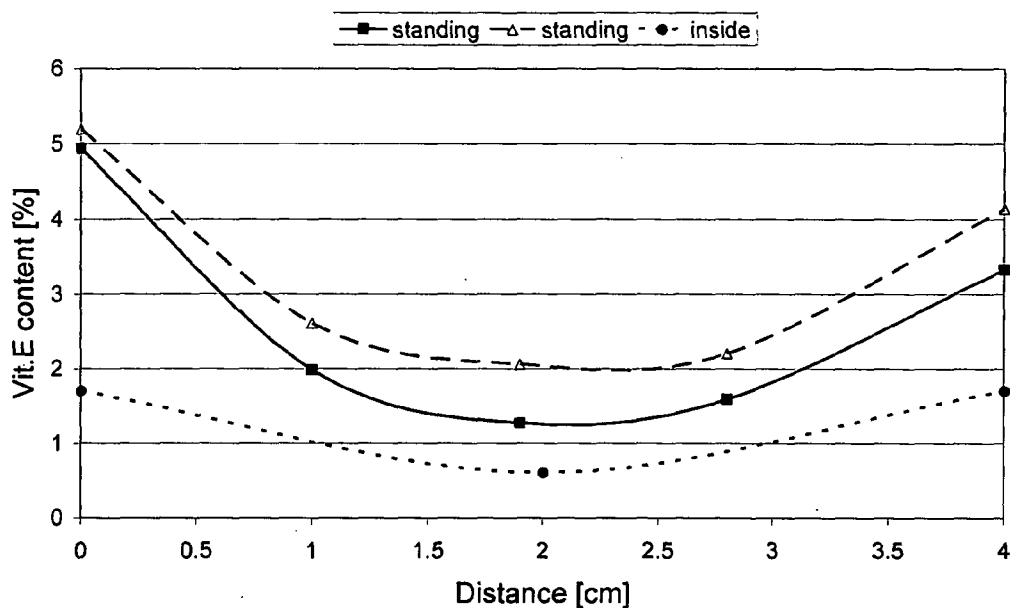


Figure 5: Vitamin E concentration profiles in the sintered blocks that were previously Compacted and soaked in a Vitamin E-hexane solution. 2 blocks were standing in the solution, partially immersed in the fluid and 1 block was completely immersed in the fluid (inside).

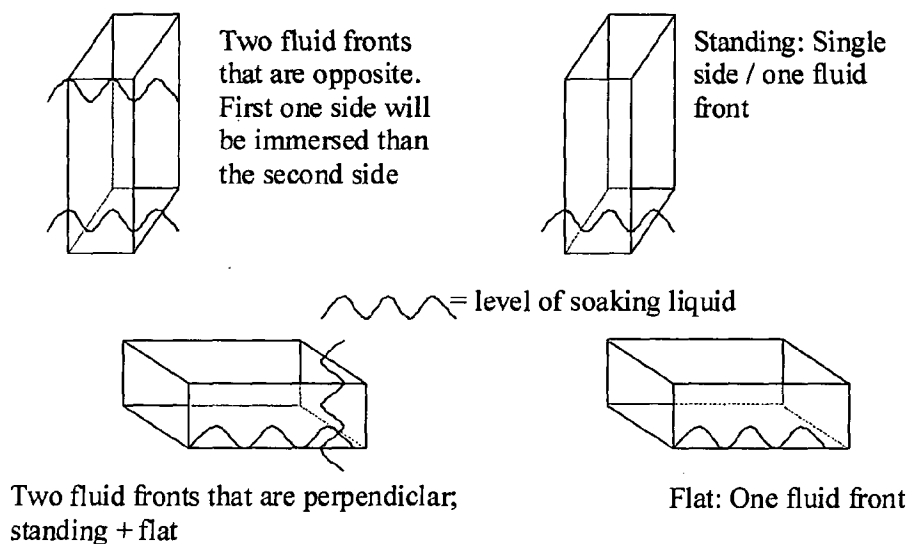


Figure 6: Examples of one or more soaking steps with different soaking directions.

2016202370 14 Apr 2016

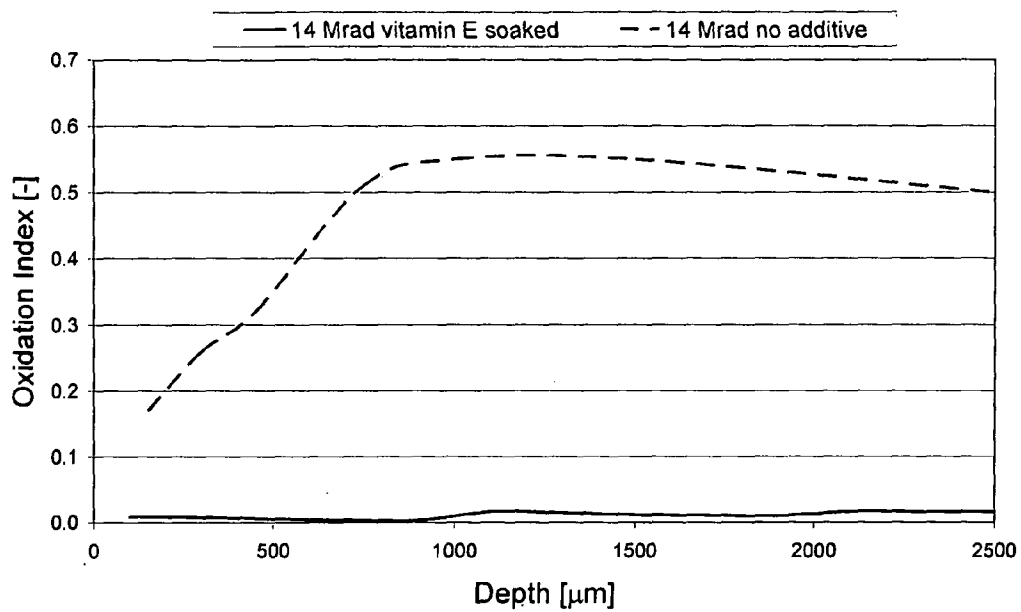


Figure 7: Oxidation profiles of vitamin E soaked and additive-free samples, both gamma irradiated with a dose of 14 Mrad in air (no post-irradiation thermal treatment).