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CA 2289762 C 2003/04/29

(11)(21) **2 289 762**

(12) **BREVET CANADIEN
CANADIAN PATENT**

(13) **C**

(86) Date de dépôt PCT/PCT Filing Date: 1998/05/04

(87) Date publication PCT/PCT Publication Date: 1998/11/26

(45) Date de délivrance/Issue Date: 2003/04/29

(85) Entrée phase nationale/National Entry: 1999/11/15

(86) N° demande PCT/PCT Application No.: EP 1998/002789

(87) N° publication PCT/PCT Publication No.: 1998/053038

(30) Priorités/Priorities: 1997/05/16 (9710048.1) GB;
1997/12/19 (9726972.4) GB

(51) Cl.Int.⁷/Int.Cl.⁷ C11D 17/00, C11D 13/18, C11D 13/16

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(54) Titre : PROCEDE DE PRODUCTION D'UNE COMPOSITION DETERGENTE

(54) Title: PROCESS FOR THE PRODUCTION OF A DETERGENT COMPOSITION

(57) Abrégé/Abstract:

A process for forming detergent bars comprising applying pressure to a detergent composition contained within a mould cavity. The detergent composition in the mould is in a substantially fluid or semi-solid state and the mould is substantially full before the pressure is applied.



PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D 17/00, 13/16, 13/18	A1	(11) International Publication Number: WO 98/53038 (43) International Publication Date: 26 November 1998 (26.11.98)
(21) International Application Number: PCT/EP98/02789 (22) International Filing Date: 4 May 1998 (04.05.98) (30) Priority Data: 9710048.1 16 May 1997 (16.05.97) GB 9726972.4 19 December 1997 (19.12.97) GB (71) Applicant (for AU BB CA CY GB GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC [GB/GB]; Unilever, Blackfriars, London EC4P 4BQ (GB). (71) Applicant (for all designated States except AU BB CA CY GB GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL).		(72) Inventors: ALLAN, Peter, Stewart; Meriton, Nicol End, Chalfont St Peter, Buckinghamshire SL9 9LU (GB). CORDELL, John, Martin; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB). IRVING, Graeme, Neil; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB). NADAKATTI, Suresh, Murigeppa; Hindustan Lever Ltd., Research Centre, 64 Main Road, Whitefield P.O., Bangalore 560 066 (IN). NAIK, Vijay, Mukund; Hindustan Lever Ltd., Research Centre, 64 Main Road, Whitefield P.O., Bangalore 560 066 (IN). OVERTON, Christine, Ann; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB). <u>STOCKER, Frederick, Edmund</u> ; Unilever Research Port Sunlight, Quarry Road East, Bebington, Wirral, Merseyside L63 3JW (GB). TARVERDI, Kamik; 4 Manor Road, Harrow, Middlesex HA1 2PB (GB). (74) Agent: MOLE, Peter, Geoffrey; Unilever plc, Patent Division, Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: PROCESS FOR THE PRODUCTION OF A DETERGENT COMPOSITION (57) Abstract A process for forming detergent bars comprising applying pressure to a detergent composition contained within a mould cavity. The detergent composition in the mould is in a substantially fluid or semi-solid state and the mould is substantially full before the pressure is applied.		

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PROCESS FOR THE PRODUCTION OF A DETERGENT COMPOSITION**TECHNICAL FIELD**

5 The present invention relates to a process for forming detergent bars and detergent bars formed thereby. The detergent bars may be intended for personal wash or fabric wash.

BACKGROUND

10 One of the conventional methods of detergent bar manufacture is casting. In the casting process, a detergent composition in a heated, mobile and readily pourable state is introduced into the top of an enclosed cavity of the desired shape within a mould and the temperature of the composition is
15 reduced until it solidifies either totally or partially, such that the bar is handleable. The mould is generally two-part, and the bar can be removed by opening the mould.

In order to be castable, the detergent formulation must be
20 mobile and readily pourable at the elevated temperatures employed. The detergent melt, once in the mould, tends to cool slowly and unevenly. This can lead to unwanted structural orientations and segregation of ingredients.

25 Detergent formulations may be desired to be formed into bars in such a condition that they are not sufficiently fluid to be cast by conventional means. In this case, pressure can be used to deliver the detergent composition to the mould in, for example, an injection moulding process. The
30 detergent composition will still require some further cooling and solidification within the mould cavity.

A major problem with such processes is that detergent compositions shrink in the mould as they cool and solidify.
35 This is highly undesirable as the mould cavity is intended

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to impart a distinctive shape on the bar and often also a logo of some kind. Shrinkage can result in dimples, wrinkles or voids in the bar, or a depression at the fill point. Which of these bar imperfections occurs depends on factors such as the nature of the detergent composition at fill, the cooling mechanism employed, and the form and surface of the mould.

A further problem particularly associated with the delivery of semi-solid detergent compositions to a mould, is the formation of "weld lines" in the bars. Without being limited by theory, it is believed that as a material is delivered to a mould cavity, flow fronts of the material are created in the cavity as material is added and the cavity fills. Weld lines in the final bars are a result of interfaces between flow fronts of detergent material inside the mould cavity which have not blended together. Such weld lines may be visible to the consumer, and can lead to weaknesses in the bar, which may in turn lead to cracking in use.

Therefore, there is a need for a process for forming detergent compositions into good quality bars which have good appearance and physical characteristics. Such a process should prevent the imperfections so far associated with shrinkage, and, should allow the detergent composition to be fed to the mould in any desired physical state, and the bar formed to be any desired shape, with a well-defined logo if required.

WO96/25481 (Unilever) and US 2,813,078 (American Cynamid) disclose processes for preparing solid blocks or tablets of detergent compositions by compressing granular or pelletised material, respectively in a moulding press.

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US 2987484 (Procter & Gamble) discloses a closed die moulding process in which a heated non-soap fluid melt mixture of synthetic detergent and a binder-vehicle is rapidly injected through a small orifice into a substantially closed die, the
5 fluid mixture being capable of solidifying into a shape-sustaining form.

The present inventors have found that if a pressure is applied to a detergent composition in a substantially fluid
10 or semi-solid state contained within a mould cavity then the problems associated with shrinkage of the detergent composition as it cools can be reduced.

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Furthermore, the problems encountered when forming bars from semi-solid material, e.g. weld lines, can be alleviated and better logo definition can be obtained.

5 SUMMARY OF THE INVENTION

According to a first aspect, the present invention provides a process for forming detergent bars comprising applying pressure to a detergent composition contained within a mould cavity characterised in that the mould cavity is
10 substantially full when the pressure is applied and the detergent composition is in a substantially fluid or semi-solid state when the pressure is first applied.

In order to maintain the total volume of detergent material
15 in the mould cavity and improve shape reproduction, further detergent composition may be delivered to the mould cavity as the volume of detergent composition in the mould cavity decreases as it cools and shrinks.

20 Thus, according to a second aspect, the present invention provides a process for forming detergent bars comprising feeding a detergent composition into a mould cavity until it is substantially full and then creating a pressure on the detergent composition in the mould cavity by applying a
25 pressure to a feed of a detergent composition in fluid contact with the material contained within the cavity.

If the detergent composition being delivered to a mould contains a quantity of a compressible material, e.g. a gas,
30 then when a pressure is applied to the detergent composition contained within the mould, the volume of the compressible material will reduce. If the mould cavity is then closed, subsequent volume reduction of the detergent material

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(e.g. shrinkage on cooling) enables the compressible material volume to increase, thus maintaining the overall shape and volume of the bar.

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Thus, according to a further aspect, the present invention provides a process for forming detergent bars comprising applying a pressure to a detergent composition contained within a mould cavity characterised in that the detergent
5 composition contains a compressible gas component dispersed therein.

Unless specified more generally, references herein to the invention or to any preferred features apply to all aspects
10 of the invention.

DETAILED DESCRIPTION OF THE INVENTION

Detergent compositions to be delivered to the mould can be in any form capable of being delivered to the mould. For
15 example, the composition may be in a substantially fluid (e.g. molten, molten dispersion, liquid), substantially semi-solid or almost solid form, so long as the composition is sufficiently plastic to allow the material to be delivered to the mould as would be understood by the person
20 skilled in the art.

The present invention envisages that detergent compositions which are not readily mobile and pourable (i.e. which are not conducive to casting) can be fed into a mould and formed
25 into bars by techniques such as injection moulding.

Preferably, the detergent composition entering the mould cavity is in a substantially fluid or semi-solid state. A detergent composition may be considered to be in a semi-
30 solid state if sufficient structure is present in the composition so that it no longer behaves like a simple liquid, as would be understood by the person skilled in the art. The term "melt" as used hereinafter, can include detergent compositions in a substantially fluid or semi-
35 solid state.

Where the pressure applied to the mould is applied over a small cross-sectional area, e.g. at the gate of the mould, it will be clear to the skilled person that the benefits of the present invention will not be obtained if the material
5 in the mould cavity is allowed to solidify to too great an extent before a pressure is applied. Hence, the material in the mould is in a substantially fluid or semi-solid state when the pressure is first applied.

10 The material should be such that it is capable of transmitting pressure applied for example at the gate of the mould cavity, through the material in the cavity.

Preferably, pressure is applied to the detergent material
15 contained within the mould cavity whilst at least part of the detergent material therein is solidifying.

The mould is substantially full when the pressure is applied.

20

Moulds used in detergent bar manufacture typically comprise a cavity defined by a rigid material. Typically, a mould comprises two (or more) rigid complementary die parts which on engagement define a cavity corresponding to the total
25 shape of the article to be manufactured in a casting or injection moulding process and a gate. The detergent melt is fed to the mould cavity through an orifice or "gate" in the mould. The gate opens on one side to the cavity and may open on the other side to a neck (e.g. a small channel)
30 through which detergent composition can be fed to the mould cavity. The neck may be designed to hold a relatively large quantity of detergent material, e.g. up to 20% of the mould cavity volume, if desired.

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Moulds are generally constructed of materials with high thermal conductivity, e.g. aluminium. This is to aid cooling of the melt and hence shorten bar release times. The mould will be designed so that it can withstand the
5 envisaged applied pressures without leakage, e.g. by provision of suitable sealing means such as "lips" and grooves, gaskets etc. between the mould parts. The mould will also be capable of being sealed at the gate, either by contact with the means for delivering detergent material, or
10 by means of a separate closure device such as a valve or shutter.

In a conventional casting process, a detergent melt in a substantially fluid state and generally at a high
15 temperature is fed to a mould until the mould cavity is full. If a detergent composition to be delivered is not in a fluid form, pressure may be applied to the detergent composition in order to deliver it to the mould cavity.

20 Once within the mould cavity, the detergent material is cooled, and this tends to result in a volume reduction of the detergent material. This may result in any of the following occurring: the detergent material pulls away from the mould wall, often preferentially over one part of the
25 mould surface; the detergent material shrinks away from an internal imperfection(s) in the bar, leaving a "hole" or void(s) within the bar; the detergent material shrinks at the gate, leaving a depression there (a "sink mark"). Loss of contact with the mould wall is visible as dimples or
30 wrinkles on the bar surface. Any depression at the gate is also readily observed. The presence of internal bar imperfections can be demonstrated non-intrusively by transmission X-ray techniques.

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A problem particularly associated with the delivery of semi-solid detergent compositions to a mould, is the formation of "weld lines" in the bars. Without being limited by theory, it is believed that as a material is delivered to a mould cavity, flow fronts of the material are created in the cavity as material is added and the cavity fills. Weld lines in the final bars are a result of interfaces between flow fronts of detergent material inside the mould cavity which have not blended together properly. Such weld lines may be visible to the consumer, and can lead to weaknesses in the bar, which may in turn lead to cracking in use. These weld lines are also detectable by X-ray techniques.

The mould can be filled up to the gate (i.e. only the mould cavity can be filled) or more material may be added which will sit, for example, in the neck of the mould.

Where a mould cavity is filled and then immediately disconnected from the feeding means any of the aforementioned phenomena can occur.

If material at the entry to the mould cavity, or in the neck of the mould solidifies quickly, subsequent shrinkage of material within the cavity may result in internal voids and/or surface effects. If the point of entry to the mould cavity remains molten, depressions/imperfections at the position of the gate are likely.

According to the process of this invention, a pressure is applied to the detergent composition contained within the mould cavity, once the cavity is full. The pressure may be applied by any suitable device, e.g. a hydraulically or pneumatically driven piston or plunger.

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In a preferred embodiment, a pressure can be applied to a detergent composition feed in fluid contact with the detergent composition in the mould cavity in order to force more material into the mould cavity and thus put the detergent composition in the mould cavity under pressure. The mould cavity can then be sealed or closed and the material contained therein allowed to cool and solidify.

The feed of detergent material to which a pressure is applied may be the same or a different detergent composition to that which is contained within the mould cavity.

Instead of closing or sealing the mould or mould cavity once a pressure has been created on the material therein, the pressure can continue to be applied on the detergent composition in the mould cavity by continuing to apply pressure to a detergent composition feed in fluid contact with the detergent composition contained within the mould cavity. Continuing to apply pressure to a detergent composition feed may mean maintaining the existing pressure on the material in the mould cavity or even increasing it.

Of course, if pressure is being applied to a detergent feed in order to create a pressure on the material contained within a mould cavity, then the detergent feed must be capable of transmitting that pressure, i.e. it is preferably in a fluid or semi-solid state. In this respect, it may be necessary to keep the detergent feed above a minimum temperature by heating it. Of course any heating means present should not interfere to any significant degree with the solidification process taking place in the mould, i.e. itcc should not heat the mould or the material in the mould to any significant degree, but only the feed outside the mould.

35

- In a preferred embodiment, a detergent melt is fed to a mould comprising a cavity, a gate and a receptacle for detergent composition on the other side of the gate (e.g. a neck or reservoir), so that the cavity is full and there is some
- 5 "surplus" detergent composition in the receptacle. A pressure is subsequently applied to the detergent composition in the receptacle using a suitable pressure applying means, such as, for example, a plunger or piston.
- 10 In the case of an injection moulding process where a pressure is applied to a feed of detergent composition in order to feed it into a mould, the pressure applied to the feed of detergent composition to deliver it to the mould cavity can continue to be applied to the feed of detergent
- 15 composition after the mould cavity has been filled.

The pressure created in the mould cavity by the processes of the present invention may be high. For example, such pressures may be up to 1000 p.s.i. (6.89 MPa). Typically,

20 pressures from 50-800 p.s.i. (0.34-5.52 MPa) can be employed. The pressure need not be constant over the time for which it is applied, i.e. it may increase or decrease.

All pressure figures are p.s.i. gauge (psig), i.e. the level

25 above atmospheric pressure.

The time over which the pressure is applied to the detergent composition after the mould cavity has been filled will vary depending on, amongst other things, the properties of the

30 detergent composition being delivered to the mould, and the cooling regime employed. For example, compositions being delivered to a mould in a substantially fluid state and at high temperatures may need a longer time than compositions which are delivered to a mould in a substantially semi-solid

35 state and/or at a lower temperature.

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Typically, the time is less than 2 minutes, preferably less than 1 minute, more preferably less than 30 seconds, and most preferably less than 10 seconds. The time may be very short, for example, less than 1 second.

5

The mould may be sealed whilst the pressure is being applied, for example by a valve or shutter, or the pressure may be applied for a period at least long enough to allow the material at the point of entry into the mould (e.g. the
10 gate) to substantially solidify, i.e. to allow a seal to form at the gate so that any further pressure applied would no longer be transmitted to the material within the mould cavity.

15 Without being limited by theory, it is believed that when a detergent composition enters a mould cavity, the material closest to the walls of the mould cavity will cool and solidify more quickly than material in the centre of the mould cavity. This results in the formation of a "shell"
20 around a more molten core. If the temperature of the mould is lower than the melt temperature when the mould is filled, this shell forms very quickly.

As there is no cool metal wall at the gate, the shell will
25 tend to be thinner, incomplete or not even present there. Once the "wall shell" has formed, and is rigid relative to the remainder of the melt, further reduction in volume will therefore tend to act at the gate. Melt at the gate will be drawn into the mould cavity. If molten material is
30 available outside the mould cavity at the gate, e.g. in a neck, this will be drawn into the mould cavity.

Another method by which a pressure can be created on the material in the mould cavity is by sealing or closing the
35 cavity after it has been substantially filled, and then

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reducing the volume of the cavity. This could be achieved, for example, by moving a wall of the mould cavity, or part thereof, e.g. the logo on the inside of the mould could be moved further into the cavity. The latter method has the
5 added advantage that it can improve the logo definition. In an alternative to sealing or closing the mould after filling, an external pressure could be maintained on the detergent composition in the cavity at any exit/entry point from the mould cavity, such as the gate, in order to
10 maintain or even increase the pressure on the material contained within the cavity as the cavity volume is reduced.

The present invention also provides for partially filling a mould cavity with a detergent composition and then reducing
15 the volume of the mould cavity until the detergent composition fills the volume of the cavity and a pressure is created on the material therein.

In a preferred embodiment, the detergent composition to be
20 formed into bars contains a compressible component or components, such as, for example a gas. The detergent composition may be aerated or there may be gas present as a result of a manufacturing process (e.g. a mixing step or as a result of a chemical reaction). The volume of gas at
25 ambient conditions may be sufficient to create floating bars, for example, or may be small enough as to not be detectable by the consumer, e.g. less than 5% by volume, preferably less than 2% by volume. The detergent composition containing a compressible gas component is
30 delivered to a mould cavity until the cavity is substantially full and a pressure is then applied to a detergent composition feed in fluid contact with the detergent composition in the mould in order to force more material into the mould cavity. The compressible component
35 (i.e. the gas) in the detergent composition in the mould

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cavity will be compressed and pressurised and more detergent material will enter the mould cavity. The mould can then either be sealed or closed or the pressure can be maintained on the detergent composition in fluid contact with the material inside the mould cavity for a period of time, as described previously. As the "non-compressible" material inside the cavity cools and decreases in volume, the volume occupied by the compressible component will increase.

10 In another aspect, the present invention provide a detergent bar obtainable by the process of the present invention.

By "detergent bar" is meant a solid, shaped object such as a tablet, cake or bar in which the level of surface active agent, which comprises soap, synthetic detergent active or a mixture thereof, is at least 5% by weight based on the bar. The detergent bar may also comprise benefit agents for imparting or maintaining desirable properties for the skin. For example, moisturising agents may be included.

20 The detergent compositions may comprise homogeneous components or mixtures of components, or may comprise material suspended or dispersed in a continuous phase.

25 **EXAMPLES**

Example

Detergent formulation A was formed into detergent bars by an injection moulding process. The detergent composition was fed into a mould cavity by means of an injection device comprising an injection chamber, an actuator, and a nozzle.

The detergent composition A was in a semi-solid state when entering the mould cavity, at a temperature of 50-55 C. In all the runs, the mould was at ambient temperature before

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fill and cooling was effected by packing solid CO₂ around the outside of the mould for the period of time specified plus maintaining the mould at ambient temperature for a further

5 5 minutes.

Formulation A was as follows:		wt % active
	Directly esterified fatty isethionate	27.8
	Sodium stearate	14.6
10	Propylene glycol	17.8
	Stearic acid	12.8
	PEG 8000	9.7
	Coco amido propyl betaine	4.9
	Paraffin wax	2.9
15	Sodium isethionate	0.4
	Water	5.6
	Minor additives (preservatives,perfume,colour etc)	2.5
	TOTAL	100.0

20 The "hold time" referred to in Table 1 is the period of time over which pressure continued to be applied to the feed of detergent composition in the injection chamber after the mould cavity had been filled. After the hold time had elapsed, the mould was disconnected from the feed means.

25

The pressure measured in the injection chamber at a point just above the entry to the nozzle is recorded as the maximum injection pressure in Table 1. The average pressure inside the mould cavity (i.e. at a side wall) over the "hold
30 time" period is recorded as the average mould pressure in Table 1.

These runs illustrate that the surface quality of the bars can be improved by applying a pressure to a detergent
35 composition in a mould. No weld lines were observed in any of the bars.

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TABLE 1

Run	Fill temp (°C)	Mould vol (g)	Cooling solid CO2 (mins)	Ease of release	Hold time (s)	Aver mould press (psig)	Max inject press (psig)	Appearance
1	53	125	0.5	Slight adhesion to one side	0.25	206 (1.42 MPa)	206 (1.42 MPa)	Dimpled, mainly on one sided
2	53	125	0.5	Easy	1	150 (1.03 MPa)	260 (1.79 MPa)	Very slightly dimpled
3	52	125	0.5	Easy	6	155 (1.07 MPa)	204 (1.41 MPa)	No dimples; very good surface
4	53	125	0.5	Easy	6	165 (1.14 MPa)	234 (1.61 MPa)	No dimples; very good surface

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CLAIMS

1. A process for forming detergent bars comprising
5 applying pressure to a detergent composition contained
within a mould cavity characterised in that the mould
cavity is substantially full when the pressure is
applied and the detergent composition is in a
substantially fluid or semi-solid state when the
pressure is first applied.
10
2. A process according to claim 1 in which pressure is
applied to the detergent composition contained within
the mould cavity whilst at least part of the detergent
composition is solidifying.
15
3. A process according to claim 1 or claim 2 in which the
pressure on the detergent composition contained within
the mould cavity is created by applying a pressure to a
feed of a detergent composition, said feed of detergent
20 composition being in fluid contact with the material
inside the cavity.
4. A process according to any one of claims 1 to 3 in
which the detergent composition contained within the mould
25 cavity contains a compressible gas component dispersed
therein.
5. A process according to any one of claims 1 to 4 in which
the mould is sealed whilst the pressure is being applied.

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6. A process according to any one of claims 1 to 5 in which the time over which the pressure is applied to the detergent composition contained within the mould cavity is less than 2 minutes.

5