

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
28 December 2006 (28.12.2006)

PCT

(10) International Publication Number  
WO 2006/136197 A1

- (51) International Patent Classification:  
A24D 3/06 (2006.01) A24D 3/04 (2006.01)
- (21) International Application Number:  
PCT/EP2005/008503
- (22) International Filing Date: 21 June 2005 (21.06.2005)
- (25) Filing Language: English
- (26) Publication Language: English
- (71) Applicant (for all designated States except US): V. MANE  
FILS [FR/FR]; 620, route de Grasse, F-06620 Bar-sur-  
Loup (FR).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): HARTMANN, Di-  
dier [FR/FR]; 83, avenue Maurice Chevalier, Résidence La  
Boissière, Bât Bambou, F-06150 Cannes La Bocca (FR).  
HANNETEL, Jean-Michel [FR/FR]; 6, rue Jeanne Jugan,  
F-06130 Grasse (FR). COURSIERES, Nathalie [FR/FR];  
580 chemin de la Stèle, G-06530 St Cezaire sur Siagne  
(FR).
- (74) Agents: TOUATI, Catherine et al.; Cabinet Plasseraud,  
52, rue de la Victoire, F-75440 Paris Cedex 09 (FR).

- (81) Designated States (unless otherwise indicated, for every  
kind of national protection available): AE, AG, AL, AM,  
AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN,  
CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI,  
GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE,  
KG, KM, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA,  
MD, MG, MK, MN, MW, MX, MZ, NA, NG, NI, NO, NZ,  
OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL,  
SM, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC,  
VN, YU, ZA, ZM, ZW.
- (84) Designated States (unless otherwise indicated, for every  
kind of regional protection available): ARIPO (BW, GH,  
GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM,  
ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM),  
European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI,  
FR, GB, GR, HU, IE, IS, IT, LT, LU, MC, NL, PL, PT, RO,  
SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN,  
GQ, GW, ML, MR, NE, SN, TD, TG).

**Declaration under Rule 4.17:**

— of inventorship (Rule 4.17(iv))

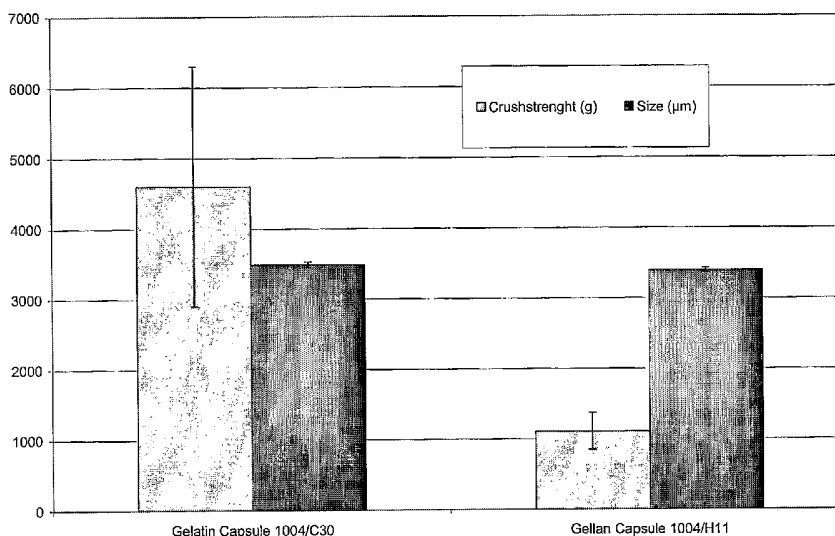
**Published:**

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: SMOKING DEVICE INCORPORATING A BREAKABLE CAPSULE, BREAKABLE CAPSULE AND PROCESS FOR MANUFACTURING SAID CAPSULE

Comparison of standard gelatin crushstrength & gellan type capsule crushstrength



(57) Abstract: A smoking device comprising a recipient including or able to receive burning products, preferably tobacco, and a filter element connected to the recipient, wherein said filter comprises at least one breakable capsule, said capsule having a initial crush strength from 0.5 to 2.5 kp, and keeping a crush strength from 0.5 to 2.5 kp and a deformation of less than two third of its diameter prior to rupture after having been submitted to a smoking test. The invention is also relating to the capsule suitable for being incorporated in a smoking device, and to the process of manufacture of said capsule.

WO 2006/136197 A1

**SMOKING DEVICE INCORPORATING A BREAKABLE CAPSULE, BREAKABLE  
CAPSULE AND PROCESS FOR MANUFACTURING SAID CAPSULE**

This invention relates to a smoking device  
5 incorporating a breakable capsule, more particularly to a  
filtered smoking device incorporating such a capsule in its  
filter element.

In the prior art, some smoking devices  
incorporating a breakable capsules are known. For example,  
10 US2004/0261807 describes a cigarette comprising a tobacco rod  
and a filter element connected to the tobacco rod, said  
filter having a cavity wherein a capsule is disposed. Said  
capsule comprises an outer gelatin shell, and an inner liquid  
composition including flavoring agents.

15 The goal of US2004/0261807 is to make it possible  
for the smoker to break the capsule, during the combustion of  
the cigarette, to allow the release of the content stored in  
the capsule.

The gelatin capsule is a good storage capsule, but  
20 is sensitive to moisture, and may soften during the smoking  
time. Because of this softening, the gelatin shell loses its  
breakability, i.e. its ability of being ruptured under the  
pressure imposed by the smoker's fingers when the smoker  
wants to release the content of the capsule.

25 The ability to rupture is measured through the  
crush strength to be exerted to rupture the capsule and  
through the deformation of the capsule when pressure is  
applied.

It is to be noted that said crush strength must not  
30 be too high at a dry state to be suitable in such an  
application. As a matter of fact, a crush strength of more  
than 2.5 kp appears too high to achieve the expected results.

Typically, the gelatin capsule of the prior art has a very important deformation when placed in smoking moisture conditions, which prevents the smoker from breaking it through a pressure of his/her fingers.

5           There is therefore a need to build up new capsules which present a crush strength of at most 2.5kp and which keep their breakability even when exposed to the moisture brought into the filter during the smoking.

10           The Applicant has now found that the incorporation of selected hydrocolloids in the outer shell of the capsule, and/or the coating of the outer shell by a moisture barrier layer, result in capsules keeping their ability to rupture within a smoking device even after exposition to the moisture brought in the smoking device by the smoker.

15           Thus, the invention relates to a smoking device comprising:

-a recipient including or able to receive burning products, preferably tobacco,

20           - a filter element which is connected to the recipient,

          wherein said filter comprises at least one breakable capsule, said capsule

- having a crush strength  $C_i$  of 0.5 to 2.5 kp,

25           - keeping a crush strength  $C_f$  of 0.5 to 2.5 kp and presenting a deformation of less than two third of its diameter prior to rupture after having been submitted to a smoking test A. (It is known from one skilled in the art that 1 kp is 9.8 N).

30           The recipient may be the rod of a cigarette, wherein the burning product is tobacco.

          The initial crush strength  $C_i$  of the capsule is measured before smoking, by continuously applying a load

vertically onto one particle until rupture using a LLOYD -  
CHATILLON Digital Force Gauge, Model DFIS 50, having a  
capacity of 25Kg, a resolution of 0.02 Kg, and an accuracy of  
+/- 0,15 %. The force gauge is attached to a stand; the  
5 capsule is positioned in the middle of a plate that is moved  
up with a manual thread screw device. Pressure is then  
applied manually and the gauge records the maximum force  
applied at the very moment of the rupture of the capsule,  
(measured in Kg or in Lb). Rupture of the capsule results in  
10 the release of the core.

The smoking test A is performed on the smoking  
Machine HEINR BORGWALDT RM 4/cs. The cigarette containing  
breakable capsule is positioned on the smoking machine as  
standard position used for other type of analysis. The  
15 adjustable parameters are set up as follows :

Puff volume: 35ml (as defined in internationally standard  
method for smoking machine)

Puff period : 60 s

Puff duration : 2 s

20 Puff sweep time : 1.8s

Exhaust sweep time : 1s

The test is finished when the cigarette is completely  
smoked or after 7 or 8 puffs.

**The final crush strength Cf is measured after said smoking Test A following**  
25 **the same procedure as used for Ci.**

The deformation of the capsule is also measured  
after smoking test A, just before the rupture of the capsule.  
The deformation corresponds to the ratio between the capsule  
diameter and the width of the capsule once pressed at the  
30 very moment of the rupture.

According to one embodiment of the invention, the smoking device includes a capsule having a crush strength  $C_i$  of 0.6 to 2 kp, preferably of 0.8 to 1.2 kp and keeping a crush strength  $C_f$  in the range of 0.6 to 2 kp, preferably in the range of 0.8 to 1.2 kp when submitted to the smoking test A.

According to another embodiment of the invention, the deformation of the breakable capsule within the smoking device prior to rupture, before and after its submission to the smoking test A, is less than 2 mm, preferably less than 1 mm.

According to a preferred embodiment of the invention, the capsule is such that it claps or makes an audible "pop", when ruptured.

Advantageously, the shell thickness of the capsule is 10-500 microns, preferably 30-150 microns, more preferably 50-60 microns; the outer diameter of the capsule is in the range of 2 to 8 mm, preferably 3 to 5 mm, more preferably 3, 5 to 4 mm; the ratio diameter of the capsule / thickness of the shell is in the range of 10 to 100, preferably 50 to 70.

The shell comprises at least one hydrocolloid selected from gellan gum, agar, carrageenans, pullulan gum or modified starch, alone or as a mixture thereof or in combination with gelatin. Advantageously, the selected hydrocolloid is gellan, and the amount of gellan present in the shell is 1.5 to 50 % w/w of the total weight of the shell. According to an embodiment of the invention, the shell contains less than 80 % gelatin, preferably less than 75 % gelatin.

In an alternative embodiment of the invention, the capsule includes a moisture barrier coating. In this embodiment, the shell of the capsule is coated with at least

one moisture barrier layer comprising at least one moisture barrier agent dispersed in an organic solvent. In this embodiment, the shell can be made of any hydrocolloid, including gelatin which can even constitutes in that case the only gelling agent of the shell. But preferably, even with the presence of the hydrophobic coating, the shell comprises also an amount of gellan, or agar, or carrageenan or pullulan gum sufficient to bring a certain resistance to moisture.

10           Advantageously, the at least one moisture barrier agent is at least one hydrophobic agent selected from those suitable for confectionery or pharmaceutical products, preferably selected from the group consisting of waxes, especially carnauba wax, candelilla wax or beeswax, carbowax, 15 shellac (in alcoholic or aqueous solution), ethyl cellulose or a combination thereof. More preferably, the at least one moisture barrier agent is ethyl cellulose or a mixture of ethyl cellulose and shellac.

          According to another embodiment of the invention, 20 the hydrophobic moisture barrier agent is a filmogen gelling agent, preferably gellan gum itself.

          The shell may further comprise at least one plasticizer, which may be glycerol, sorbitol, maltitol, triacetine or polyethylene glycol, or another polyalcohol 25 with plasticizing properties, and optionally one acid of the monoacid, diacid or triacid type, in particular citric acid, fumaric acid, malic acid, etc. The shell can advantageously comprises a coloring agent which renders easier the location of the capsule in the filter during the manufacturing process 30 of filters. Fillers can also be included in the composition of the shell.

The core of the capsule included within the smoking device of the invention is preferentially composed of a mixture of materials or products which are lipophilic or partially soluble in ethanol, or of molecules formulated as  
5 oil/water/oil emulsions.

The core of a breakable capsule according to the invention represents by weight 50 to 92% of said capsule, preferably 80 to 92%, more preferably 85 to 92%.

The core of the capsule may be composed of one or  
10 more lipophilic solvents conventionally used in the food, pharmaceutical or cosmetic industries. In a preferred embodiment, these lipophilic solvents may be triglycerides, especially medium chain triglycerides, and in particular triglycerides of caprylic and capric acid, or mixtures of  
15 triglycerides such as vegetable oil, olive oil, sunflower oil, corn oil, groundnut oil, grape seed oil, wheat germ oil, mineral oils and silicone oils. The amount of lipophilic solvent in the core of a capsule according to the invention is of the order of 0.01 to 90%, preferentially 25 to 75%, of  
20 the total weight of the capsule.

The core may also comprise one or more aromatic or fragrance molecules as conventionally used in the formulation of flavoring or fragrance compositions. Mention will in particular be made of aromatic, terpenic and/or  
25 sesquiterpenic hydrocarbons, and more particularly essential oils, alcohols, aldehydes, phenols, carboxylic acids in their various forms, aromatic acetals and ethers, nitrogenous heterocycles, ketones, sulfides, disulfides and mercaptans which may be aromatic or non aromatic. It may also comprise  
30 one or more molecules or extracts for cosmetic use.

The core may also comprise one or more fillers as used in aromatic emulsions. Mention will be made of dammar

gum, wood resins of the ester gum type, sucrose acetate isobutyrate (SAIB) or brominated vegetable oils. The function of these weighting agents is to adjust the density of the liquid core.

5           The core or the shell may also comprise one or more sweeteners, which may be provided in the form of a solution or suspension in ethanol. Examples of suitable sweeteners may be, but is not limited to, aspartame, saccharine, NHDC, sucralose, acesulfame, neotame, etc.

10           The core may also comprise one or more "sensate" aromatic agents, which provide either a freshening effect or a hot effect in the mouth. Suitable freshening agents may be, but are not limited to, menthyl succinate and derivatives thereof, in particular Physcool® marketed by the Applicant. A  
15 suitable hot effect agent may be, but is not limited to, vanillyl ethyl ether.

          The flavoring agents that can be solubilized in the solvent of the core of the capsule include, but are not limited to, natural or synthetic aromas and/or fragrances.  
20 Examples of suitable fragrances are fruity, confectionery, floral, sweet, woody fragrances. Examples of suitable aromas are vanilla, coffee, chocolate, cinnamon, mint.

          The invention also relates to a breakable capsule suitable for being incorporated in the filter of a smoking  
25 device, which is substantially moisture stable. "Substantially moisture stable " means that the outer shell or outer coating of the capsule has the ability to retard degradation of the capsule by means of the water brought into the smoking device by a smoker.

30           The breakable capsule according to the invention comprises a core as described hereinbefore, and a shell, said capsule presents a crush strength from 0.5 to 2.5 kp, and



keeps a crush strength from 0.5 to 2.5 kp and a deformation of less than two third of its diameter after having been submitted to the smoking test A.

According to a preferred embodiment, the capsule of  
5 the invention is a seamless capsule, obtained trough a co-extrusion process.

All the characteristics described hereabove in relation with the consitution of the capsule of the smoking device also apply to the breakable capsules according to the  
10 invetion.

The co-extrusion process is a synchronous extrusion of two liquids: the external and hydrophilic liquid phase, and the internal and lipophilic liquid phase. The co-extrusion process consists of three main stages: compound  
15 drop formation, shell solidification and capsule collection. The compound drop is a sphere of the liquid fill phase inside the shell phase. The liquid fill phase is hereinafter referred to as "the core". The shell phase is hereinafter referred to as "the shell". The capsules of the invention may  
20 be produced by any suitable co-extrusion process. Preferably, the capsules are produced by an apparatus and a process as described in EP 513603, the disclosure of which is herein incorporated by reference.

According to an embodiment of the invention, after  
25 the co-extrusion step, the solidification step is performed by keeping cold the capsules in order to ensure correct gelling of the shell for example by contacting them with a cold bath. The cold bath is preferably cold oil.

The capsules may then be centrifuged in order to  
30 remove the surplus oil, optionally washed with organic solvent (such as acetone, ethyl acetate, ethanol, petroleum ether, etc.) also to remove the surplus oil, and dried.

According to one embodiment of the invention, after the co-extrusion step, and eventually the solidification step, the capsules are centrifuged.

According to another embodiment of the invention,  
5 the capsules are co-extruded, centrifuged and optionally immersed into a solution or an emulsion containing a curing agent.

The curing agent may also be ethanol or any other anhydrous organic solvent, such as ethyl acetate or isopropanol, maintained at a temperature of between 0 and  
10 25°C, more particularly between 10 and 20°C.

The curing agent can alternatively or also be a bath of calcium ions, for example of calcium chloride, dicalcium phosphate or calcium sulfate or a bath of acid  
15 containing calcium ions of pH less than 5, preferably of 3 to 4. Examples of acids may be adipic acid, fumaric acid, gluconic acid or glucono-delta-lactone. The calcium ion or acid bath is preferably at a temperature of 0 to 25°C, preferably 10 to 20°C.

20 The effect of the immersion step is to wash out the oil remaining at the periphery of the capsule, and to gradually strengthen the shell, notably through dehydration and osmotic equilibrium.

According to an embodiment of the invention, after  
25 immersion, the capsules are dried in a current of air at controlled temperature and humidity. The relative humidity of the drying air is 20% to 60%, preferably 30 to 50%; the temperature of the drying air is of 15 to 60 °C, preferably 35 to 45 °C.

30 According to an embodiment of the invention, the capsules can be obtained using the following method: co-extrusion of the external and hydrophilic liquid phase on the

one hand, and of the internal and lipophilic liquid phase and components of the core on the other hand, optionally centrifugation, optionally immersion of the capsules so obtained in a bath of calcium ions or acid, followed by  
5 drying.

According to another embodiment, the process according to the invention further comprises a coating step during which the moisture barrier outer layer is applied on the capsules. Preferably, said coating step is performed by  
10 dipping the capsules in a coating solution, or by spraying a coating solution onto the capsules. Said coating step is preferably performed after the drying step.

The capsules manufactured through the process of the invention are essentially or perfectly spherical and very  
15 homogeneous in size.

The invention is hereunder illustrated by the following examples, which should not be considered as limiting the scope of the invention.

20

### EXAMPLES

#### Example 1

Two types of capsules presenting the same size are prepared by co-extrusion as disclosed in patent EP 513603. The composition of capsules according to the present  
25 invention, referenced as 1004/H11 (having less than 80 % gelatin and including gellan gum) is given in Table 1 below, and the composition of prior art capsules containing 80% gelatin, referenced 1004/C30, is given in Table 2 below.

30

**Table 1 - Capsules 1004/H11**

<b>External liquid phase</b>	<b>%/total</b>	<b>%/dry matter</b>
<b>Dry matter : 15.5%</b>	<b>weight</b>	
Gellan gum Kelco F	1.800%	11.62%
Gelatin 260A	4.000%	25.82%
Sorbitol	1.000%	6.46
Glycerol	0.500%	3.23
Dextrin Cristal Tex T648	8.000%	51.65%
Sodium bicarbonate	0.180%	1.16
brillant blue FD&C#1	0.010%	0.06
processing water	84.510%	
	100.000%	100%
<b>Internal liquid phase</b>	<b>%</b>	
Ethanol	6.0000%	
Miglyol	65.5000%	
Menthol	28.5000%	
<b>Total</b>	<b>100.0000%</b>	

weight of each capsule: 20.56mg

in which weight of the shell: 3.68 mg (17,89%)

5 weight of the core: 16.88 mg (82.11%)

**Table 2 - Capsules 1004/C30**

<b>External liquid phase</b>	<b>%/total</b>	<b>%/dry matter</b>
<b>Dry matter: 24.5%</b>	<b>weight</b>	
Gelatin 260A	19.800	80.82
Sorbitol	2.7000	11.02
Caramel color	2.000	8.16

processing water	75.500	
	100.000%	100%
<b>Internal liquid phase</b>		
Ethanol	5.0000	
Miglyol 812S	87.0000	
Spearmint flavor #831661 supplied by MANE	8.0000	
<b>Total</b>	<b>100,0000%</b>	

Capsules 1004/C30 and capsules 1004/H11 present a diameter of respectively 3489 +/- 40 and 3394 +/- 35  $\mu\text{m}$ .

- 5 The crush strength of the capsules is measured as follows using a LLOYD-CHATILLON Digital Force Gauge - Model DFIS 50  
Capacity = 25 Kgf (50lb)  
Resolution = 0.02Kgf (0.05lb)  
Accuracy = +/- 0.15%
- 10 Sampling Rate = 1000 times per second

Methodology:

Force Gauge is attached to a stand.

- 15 The capsule is positioned in the middle of a plate that is moved up with a manual thread screw device. Pressure is then applied manually and once the capsule wall fails, the gauge records the maximum force applied prior to rupture (measure in Kg or in lb). Sample of 10 to 20 capsules is measured.

- 20 The results are as follows:

capsules	Crush strength (Kp)	Standard deviation
1004/C30	4.60	1.11
1004/H11	1.70	0.26

The results are also represented on Figure 1.

### Example 2

5 Capsules are prepared according to example 1.

Core content is about 89 % and shell content is about 11 %, corresponding to a thickness of dried shell of about 50 microns. Capsules are then dried to obtain 3.5 mm spherical capsules with a crush-strength of 1 kp. Average  
10 weight of capsule is about 20 mg.

A prior art capsule containing gelatin (ref 1004/C1) containing 72 % gelatin and a capsule as described in example 1 above are incorporated into a cavity filter of a cigarette.

15

Crush strength value  $C_i$  is measured for both capsules according to the herein described method.

The obtained cigarettes are then smoked on a smoking machine (smoking test A) according to international standard  
20 procedure. Filters containing the capsules are collected and capsules are extracted from the filter to measure crush strength value  $C_f$  after smoking process.

In the case of gelatin capsules, capsules have melted into  
25 the filter during the smoking process and crush strength value is not measurable. Capsules are deformed and cannot burst when press on them. Gellan-including capsule, on the contrary, still burst when pressure on the filter.

Instrumentation:

- **Smoking Machine HEINR BORGWALDT RM 4/cs**

- 5 The cigarettes containing breakable capsules are positioned on the smoking machine as standard position used for other type of analysis.

The adjustable parameters are set up as follows :

10

Puff volume : 35ml (as defined in internationally standard method for smoking machine)

Puff period : : 60 s

Puff duration : 2 s

15 Puff sweep time : 1.8s

Exhaust sweep time : 1s

Crush strength are measured according to the procedure described in example 1 above.

**Capsules 1004/C1**

<b>Internal liquid phase</b>			
Dry matter: 25,0%			
Components		%	%/dry matter
Gelling agent	Gelatin	18.000%	72.00%
Plasticizer	Sorbitol	4.000%	16.00%
filler	Calcium Carbonate	1.000%	4.00%
Color	Caramel	2.000%	8.00%
Solvent	water	75.000%	
Total		100.000%	100%

<b>Internal liquid phase</b>			
<b>Name</b>		<b>%</b>	
Solubilising agent	Ethanol	5.0000%	
carrier	Miglyol	87.0000%	
Aroma	Spearmint flavor #831661 supplied by MANE	8.0000%	
	<b>Total</b>	<b>100.0000%</b>	

weight of each capsule: 20.57 mg

in which weight of the shell: 2.37 mg (11,51%)

weight of the core: 18.20 mg (88.49%)

5

The results are as follows:

capsules	Crush strength Ci	Crush strength Cf
1004/C1	1.70kp	Not measurable (capsule has melted)
1004/H11	1.2kp	1.2kp

After smoking, prior art capsules are no longer breakable.

10 The results are also presented on Figure 2.

### Example 3

15 Capsules are prepared according to example 1, using the following composition:



**Capsules 1004/H6**

<b>External liquid phase</b>			
Dry Matter:16,5%			
<b>Components</b>		<b>%</b>	<b>%/dry matter</b>
gelling agent 1	Gellan gum	2.200%	<b>13.33%</b>
gelling agent 2	Gelatin	4.000%	<b>24.24%</b>
filler	Dextrin CT648	10.000%	<b>60.61%</b>
acid or salt	Sodium bicarbonate	0.200%	<b>1.21%</b>
Color	brillant blue FD&C #1	0.10%	<b>0.61%</b>
Solvent	Osmosed water	83.500%	
Total		100.000%	
<b><u>Internal liquid phase</u></b>			
<b>components</b>		<b>%</b>	
Solubilising agent	Ethanol	5.0000%	
carrier	Miglyol 812S	90.0000%	
aroma	Spearmint flavor	5.0000%	
	<b>Total</b>	<b>100.0000%</b>	

weight of each capsule: 20.96mg

5 in which, weight of the shell: 3.72 mg (17,74%)

weight of the core: 17.24 mg (82.26%)

One capsule was introduced in a cavity of a cigarette filter. Crush strength  $C_i$  was measured according to the procedure described in example 1 above.

10  $C_i = 0.80 \pm 0.20$  Kp

Then the cigarette was submitted to Test A as described in example 2 above, for 8 puffs.

After 8 puffs, the capsule breaks with an audible 'pop'.

CLAIMS

1. A smoking device comprising:
  - a recipient including or able to receive burning  
5 products, preferably tobacco,
  - a filter element connected to the recipient ,  
wherein said filter comprises at least one  
breakable capsule, said capsule
    - having a initial crush strength  $C_i$  from 0.5 to  
10 2.5 kp,
    - keeping a crush strength  $C_f$  from 0.5 to 2.5 kp  
and presenting a deformation of less than two third of its  
diameter prior to rupture after having been submitted to the  
smoking test A.
- 15 2. The smoking device according to claim 1, wherein the  
breakable capsule is an essentially spherical seamless  
capsule.
3. The smoking device according to anyone of claims 1 or 2,  
wherein the crush strengths  $C_i$  and  $C_f$  are comprised between  
20 0.8 to 1.2 kp.
4. The smoking device according to anyone of claims 1 to 3,  
wherein the deformation of the breakable capsule prior to  
rupture, after submission to the smoking test A, is less than  
2 mm, preferably less than 1 mm.
- 25 5. The smoking device according to anyone of claims 1 to 4,  
wherein the capsule claps when ruptured.
6. The smoking device according to anyone of claims 1 to 5,  
wherein the shell thickness of the capsule is 10-500 microns,  
preferably 30-150 microns, more preferably 50-60 microns.

7. The smoking device according to anyone of claims 1 to 6, wherein the outer diameter of the capsule is from 2 to 8 mm, preferably 3 to 5 mm, more preferably 3.5 to 4 mm.

8. The smoking device according to anyone of claims 1 to 7,  
5 wherein the ratio (diameter of the capsule / thickness of the shell) is in the range of 10 to 100.

9. The smoking device according to anyone of claims 1 to 8, wherein the breakable capsule includes, in a core surrounded by a shell, fragrances or aromas dispersed or solubilized in  
10 a medium chain triglyceride solvent.

10. The smoking device according to anyone of claims 1 to 9, wherein the capsule comprises a core and a shell, said shell comprising at least one hydrocolloid selected from gellan gum, agar, carrageenans, pullulan gum or modified starch,  
15 alone or as a mixture thereof or in combination with gelatin.

11. The smoking device according to anyone of claims 1 to 10, wherein the hydrocolloid is gellan, and the amount of gellan present in the shell is 1.5 to 50 % w/w of the shell.

12. The smoking device according to anyone of claims 1 to  
20 11, wherein the shell is coated with at least one moisture barrier outer layer.

13. The smoking device according to anyone of claims 1 to 9, wherein the shell comprises gelatin alone or in combination with at least one hydrocolloid selected from gellan gum, agar, carrageenans, pullulan gum or modified starch or a  
25 mixture thereof, said shell being coated with at least one moisture barrier outer layer.

14. The smoking device according to anyone of claims 12 to 13, wherein the moisture barrier layer comprises at least a  
30 hydrophobic agent suitable for confectionery or

pharmaceutical products, preferably selected from the group consisting of waxes, especially carnauba wax, candelilla wax or beeswax, shellac, ethylcellulose or a combination thereof.

15. The smoking device according to anyone of claims 12 to  
5 14, wherein the hydrophobic moisture barrier agent is a filmogen gelling agent, preferably gellan gum.

16. A breakable capsule suitable for being incorporated in the filter of a smoking device, comprising a core and a shell, said capsule:

10 - having an initial crush strength  $C_i$  from 0.5 to 2.5 kp and keeping a crush strength  $C_f$  from 0.5 to 2.5 kp after having been submitted to the smoking test A,

- and presenting a deformation of less than two third of its diameter prior to rupture after having been submitted  
15 to the smoking test A.

17. A breakable capsule according to claim 16, wherein the capsule is a seamless capsule.

18. A breakable capsule according to anyone of claims 16 or 17, wherein the capsule comprises a core and a shell, said  
20 shell comprising at least one hydrocolloid selected from gellan gum, agar, carraghenan, or modified starch, alone or as a mixture thereof or in combination with gelatin.

19. A breakable capsule according to claim 18, wherein the hydrocolloid is gellan, and the amount of gellan present in  
25 the shell is 1.5 to 50 % w/w of the shell.

20. A breakable capsule according to anyone of claims 16 to 19, wherein the capsule comprises a core and a shell, said shell being coated with at least one moisture barrier outer layer.

21. A breakable capsule according to anyone of claims 16 or 17, wherein the shell comprises gelatin alone or in combination with at least one hydrocolloid selected from gellan gum, agar, carraghenan, or modified starch or a  
5 mixture thereof, said shell being coated with at least one moisture barrier outer layer.

22. A breakable seamless capsule according to claim 21, wherein the moisture barrier layer comprises at least a hydrophobic agent suitable for confectionery or  
10 pharmaceutical products, preferably selected from the group consisting of waxes, especially carnauba wax, candelilla wax or beeswax, shellac, ethylcellulose or a combination thereof.

23. A breakable seamless capsule according to claim 22, wherein the hydrophobic moisture barrier agent is a filmogen  
15 gelling agent, preferably gellan gum.

24. A filter for smoking device comprising at least a breakable capsule as defined in anyone of claims 16 to 23.

25. Use of a breakable capsule according to anyone of claims 16 to 23 in a smoking device.

20 26. Process for manufacturing a breakable capsule according to anyone of claims 16 to 23, comprising:

- co-extruding an external and hydrophilic liquid phase, and an internal and lipophilic liquid phase, in order to form a capsule constituted of a core comprising the  
25 internal and lipophilic phase and a shell comprising the external and hydrophilic phase,

- solidifying and/or gellifying the surface of the shell by keeping the capsule under cold conditions,

- eventually washing the so-obtained capsule with  
30 an organic solvent,

- drying the capsule,

- optionally coating the obtained capsule with at least one moisture barrier outer layer.

27. Process for manufacturing a breakable capsule according to claim 26, wherein after the co-extrusion step,  
5 the capsules are centrifuged, after or before the solidification step.

28. Process for manufacturing a breakable capsule according to anyone of claims 26 or 27, comprising a further step of immersion of the capsules into an organic liquid or an  
10 emulsion containing a curing agent.

29. Process for manufacturing a breakable capsule according to claim 28, wherein the curing agent is a solution comprising bivalent ions, preferably calcium ions.

Comparison of standard gelatin crushstrenght & gellan type capsule crushstrenght

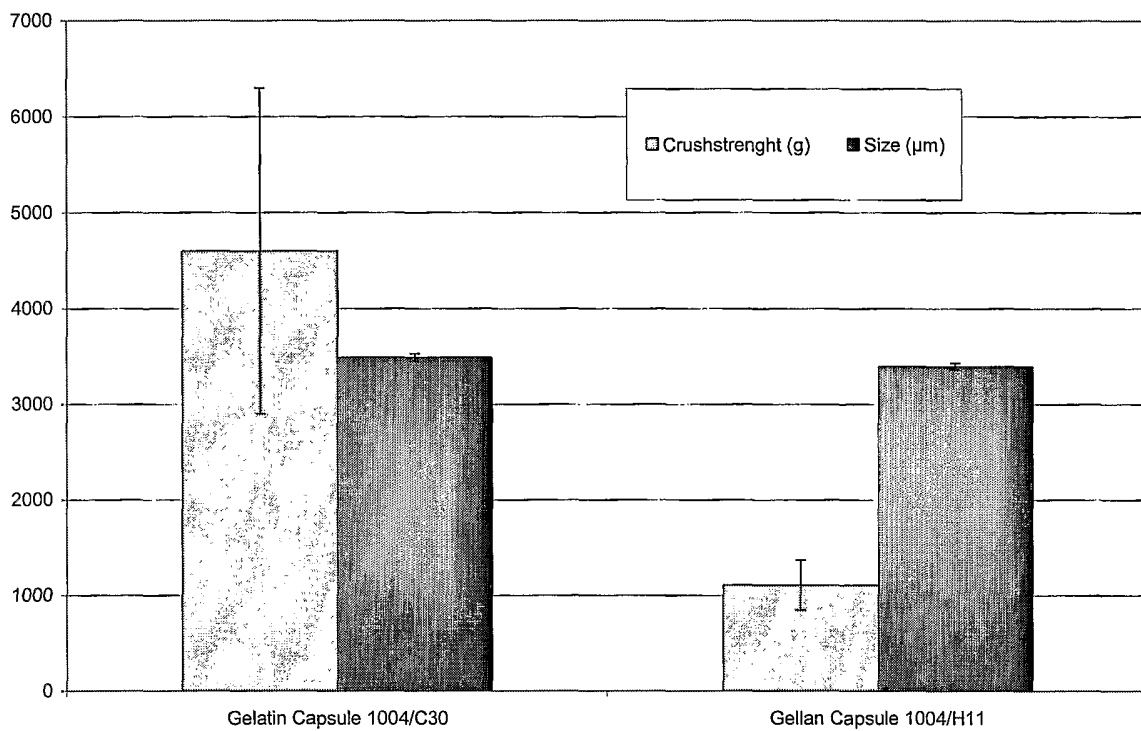


Fig. 1

Comparison of capsule crushstrenght value (Kp)  
before and after smoking

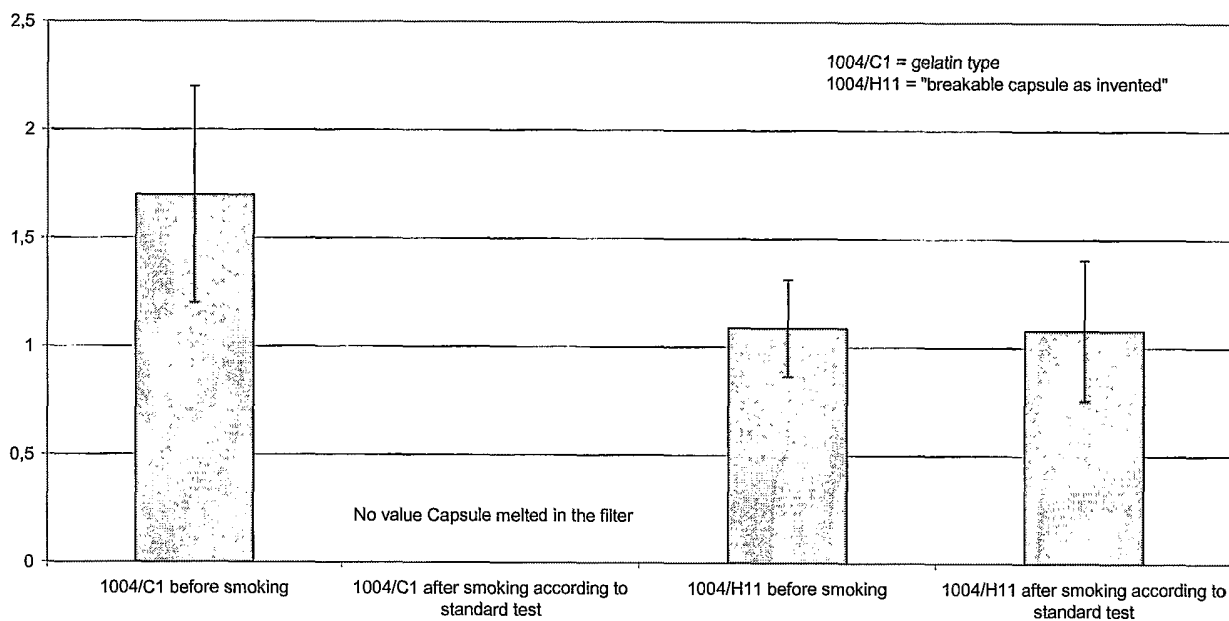


Fig.2



INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2005/008503

A. CLASSIFICATION OF SUBJECT MATTER  
INV. A24D3/06 A24D3/04

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)  
A24D

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, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 03/009711 A (KIM, JINHEE) 6 February 2003 (2003-02-06) page 6, lines 9-24 -----	1-25
A	US 2004/074507 A1 (MACADAM KEVIN GERARD ET AL) 22 April 2004 (2004-04-22) paragraph [0033] - paragraph [0034]; figures -----	1,16,25
A	US 2005/070409 A1 (DEAL PHILIP ANDREW) 31 March 2005 (2005-03-31) paragraph [0089] - paragraph [0090] -----	1,16,25
A	US 3 602 231 A (MORTIMER RUSSELL DOCK) 31 August 1971 (1971-08-31) the whole document -----	1,5,16, 25
	-/--	

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

17 January 2006

Date of mailing of the international search report

26. 05. 2006

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

MARZANO MONTEROSSO

## INTERNATIONAL SEARCH REPORT

International application No  
PCT/EP2005/008503

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 943 940 A (MINAMI ET AL) 16 March 1976 (1976-03-16) column 5, line 40 - column 6, line 5 -----	1,16,25
A	US 2004/261807 A1 (DUBE MICHAEL FRANCIS ET AL) 30 December 2004 (2004-12-30) cited in the application the whole document -----	1,16,25

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/EP2005/008503

## Box II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1.  Claims Nos.:  
because they relate to subject matter not required to be searched by this Authority, namely:
  
2.  Claims Nos.:  
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
  
3.  Claims Nos.:  
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

## Box III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1.  As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
  
2.  As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
  
3.  As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
  
4.  No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

1-25

Remark on Protest

- The additional search fees were accompanied by the applicant's protest.
- No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-25

Breakable capsule suitable for being incorporated in a smoking device, said capsule having a given initial crush strength and a given deformation after having been submitted to a smoking test.

Smoking device comprising a recipient including burning products and a filter element connected to the recipient, the filter comprising a breakable capsule as described above

---

2. claims: 26-29

Process for manufacturing a breakable capsule comprising the steps of coextrusion, solidification and drying of the capsule.

---

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No PCT/EP2005/008503
---

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
WO 03009711	A	KR 2003009800 A	05-02-2003
US 2004074507	A1	NONE	
US 2005070409	A1	WO 2005032286 A2	14-04-2005
US 3602231	A	NONE	
US 3943940	A	NONE	
US 2004261807	A1	EP 1638420 A2	29-03-2006
		WO 2005000044 A2	06-01-2005