



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/BR90/00009 <b>(22) International Filing Date:</b> 22 August 1990 (22.08.90)  <b>(30) Priority data:</b> PI 8904238                      23 August 1989 (23.08.89)                      BR  <b>(71) Applicant (for all designated States except US):</b> JOHNSON & JOHNSON INDÚSTRIA E COMÉRCIO LTDA. [BR/BR]; Rua Gerivativa no 207, Butantã, 05501-São Paulo, SP (BR).  <b>(72) Inventor; and</b> <b>(75) Inventor/Applicant (for US only) :</b> PEREIRA, José, Antonio [BR/BR]; Rua Oswaldo N. Leal no 424, Vila Rubi, 12200-São José dos Campos, SP (BR).		<b>(74) Agent:</b> DANNEMANN, SIEMSEN, BIGLER & IPANE-MA MOREIRA; Rua Marquês de Olinda no 70, Botafogo, 22251-Rio de Janeiro, RJ (BR).  <b>(81) Designated States:</b> AT, AT (European patent), AU, BB, BE (European patent), BF (OAPI patent), BG, BJ (OAPI patent), BR, CA, CF (OAPI patent), CG (OAPI patent), CH, CH (European patent), CM (OAPI patent), DE*, DE (European patent)*, DK, DK (European patent), ES, ES (European patent), FI, FR (European patent), GA (OAPI patent), GB, GB (European patent), HU, IT (European patent), JP, KP, KR, LK, LU, LU (European patent), MC, MG, ML (OAPI patent), MR (OAPI patent), MW, NL, NL (European patent), NO, RO, SD, SE, SE (European patent), SN (OAPI patent), SU, TD (OAPI patent), TG (OAPI patent), US.  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> SUPERABSORBENT MATERIAL, SUPERABSORBENT STRUCTURE, ABSORBENT ARTICLE AND PROCESS FOR PREPARING SAID SUPERABSORBENT  <b>(57) Abstract</b>  There is described a superabsorbent material having improved properties, consisting of a superabsorbent polymer whose original structure has been modified by expansion so that the final structure is expanded. This material is highly effective for the formation of absorbent structures and absorbent articles. The invention further provides a process for the preparation of said superabsorbent materials.		

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Title: "SUPERABSORBENT MATERIAL, SUPERABSORBENT STRUCTURE, ABSORBENT ARTICLE AND PROCESS FOR PREPARING SAID SUPERABSORBENT"

The present invention relates to expanded superabsorbent materials, and in particular to expanded superabsorbents for use  
5 in absorbent products such as sanitary napkins, catamenial tampons, diapers, dressings or the like, for the absorption of body fluids.

A large variety of absorbent materials are currently known, and generally used in the forming of absorbent structures. For example, absorbent materials based on cellulose pulp are well known.  
10 Another type of absorbent material well known for its great capability of absorbing liquids belongs to the class of naturally occurring absorbent materials and is known as sphagnum.

More recently, a new class of absorbent material has been developed, this class presenting a great capacity for retention  
15 and absorption of liquids. These materials are more commonly known in the art as "superabsorbents".

Much research is currently being undertaken to improve both the properties of retention and absorption of the above materials, giving emphasis to sphagnum and other superabsorbent materials as these are more directly related to the present invention.  
20

About 350 different species of sphagnum, a moss of the family sphagnaceae, are found naturally in all parts of the world, generally in acid soils subjected to flooding such as swamps, lakes and shallow lagoons. The moss forms dense and extensive colonies,  
25 its vegetative propagation occurring by ramification and death of older parts of the plant. The leaves are formed of living and dead cells. The living cells are green and are called chlorocysts, having an assimilatory function. The dead cells are colourless and are called lencocysts, and these have spiral ribs and pores which  
30 function as reservoirs for water and other aqueous liquids.

The term "liquid" used herein signifies water or any aqueous liquid to be absorbed.

Superabsorbent materials, so-called due to their large capacity for absorption and retention of liquids, are generally polymeric materials whose polymeric chains have highly hydrophilic active sites.

Superabsorbent materials may be by nature entirely synthetic, or may be obtained from essentially natural polymers in whose chains are introduced highly hydrophilic groups.

Outstanding the class of superabsorbent polymers are, inter alia, the acrylate based polymers such as sodium polyacrylate.

In addition to the examples of polymers cited above, superabsorbent polymers may include various other types of natural or synthetic polymers based on or modified by highly hydrophilic groups which are well known to those skilled in the art.

Table I below shows, by way of illustration, the liquid absorption capacities of some absorbent materials cited above, when subjected to a pressure in the range of from 0.05 to 0.5 psi. The apparatus used to evaluate this property was the GATS, with a 1% saline NaCl solution.

TABLE I

Type of Absorbent Material	Density (g/cm <sup>3</sup> )	Absorption Capacity (g/g)	
		0.05 psi	0.5 psi
Ground Pulp*	0.03	14-16	8-9
Sphagnum**	0.05	28-30	15-16
Superabsorbent*** (Favor SAB)	0.08	38-45	± 25

\* Conventional ground wood pulp used in the production of diapers and sanitary napkins.

\*\* Sphagnum sample from Botanical Institute.

\*\*\* Favor SAB sodium polyacrylate based polymer produced

by STOCKHAUSEN - Germany.

By means of a simple comparison between the values of absorption capacity listed in the above table, the superiority of superabsorbents over sphagnum and Kraft pulp is evident.

5           However, although the absorbent materials above, particularly sphagnum and superabsorbent, present satisfactory indices of absorption capacity, these materials are subjected to limitations which prevent the obtention of an absorbent material or structure truly effective for the desired purpose. For example, pulp presents  
10 a high absorption rate, but its absorption capacity and retention are low. Sphagnum, on the other hand, shows a good absorption rate and a good capacity for retention, while most superabsorbents have a very low absorption rate associated with the highest absorption and retention capacity.

15           It is well known in the art that one of the essential characteristics of an absorbent material consists in its initial absorption speed. However, it should be emphasized that as well as presenting a satisfactory absorption speed the absorbent material should simultaneously have satisfactory retention and absorption  
20 characteristics. Thus, for example, a high initial absorption rate is of little use if the capacity for retention or capacity for absorption is unsatisfactory.

25           The present invention provides a superabsorbent material with improved liquid absorption properties and free of the disadvantages shown by absorbent materials of improved properties herein described.

          A third aspect of the present invention is the provision of new absorbent articles including the absorbent structures cited above.

30           Additionally the present invention deals with a process for the obtention of the said improved superabsorbent materials.

          More specifically, the present invention reveals a new superabsorbent material obtained from a superabsorbent material commercially available, which presents a combination of the effective characteristics of the starting superabsorbent without simultaneously presenting the disadvantages shown thereby.  
35

          The present invention is prompted by the observation of the following two aspects:

1. Functional mechanism of natural sphagnum, pulp and of

superabsorbent materials.

The mechanism of absorption of natural sphagnum is by a purely physical process. The structure of sphagnum is microporous, the micropores being principally responsible for its absorptive properties. This microporous structure functions by capillary action, providing penetrating paths for the liquid to be absorbed. The liquid is stored in the reservoir cells present in the structure of sphagnum.

The mechanism of absorption of pulp or other cellulosic material is very similar to sphagnum, except that the pores are limited to the void volume between the fibres. The resulting pores are much larger than those of sphagnum, and as a consequence the capillary action and retention are much less pronounced.

The absorption mechanism of superabsorbent materials is entirely different, occurring by an essentially chemical process. The highly hydrophilic groups present in the polymeric chains of superabsorbents are essentially those responsible for its high absorption and retention capacity. The liquid to be absorbed attaches itself chemically to the hydrophilic groups present in the polymeric structure, by means of electrostatic forces of the hydrogen bridge type.

2. Limitative factors of the absorbent materials defined above:

The absence of a chemical absorption mechanism in the sphagnum or pulp structure results not only in a smaller capacity for absorption of liquids, but also reduces the retention capacity for liquids since the material absorbed is simply stored in reservoir cells in the structure without being chemically attached to the material thereof. When subject to pressure, the absorbed liquid is readily expelled from the reservoir cells according to the pressure applied.

Notwithstanding the fact that the hydrophilic groups present in the polymeric chains of superabsorbent materials make them extremely efficient in their capacity for absorbing and retaining liquids, these materials are generally restricted in their absorption and distribution speeds.

As liquids are absorbed into the structure of a superabsorbent, a "blocking action" to the later absorbed liquid is absorbed. This diminishes not only the absorption speed, but also the

capacity for distributing liquid through the structure. It has been seen that the disadvantages occur due to the fact that a structure of the superabsorbent is an extremely compact structure.

Thus the material aspect of the present invention resides in the fact that the properties of the superabsorbent material may be greatly improved by the modification of the original compact structure to a structure having micropores, such as those which exist in the structure of natural sphagnum.

As a result of this modification, there is obtained an expanded superabsorbent material having a high capacity for absorption and a satisfactory retention of liquids, without simultaneously presenting the limitations on the distribution speed caused by the "blocking action". The superabsorbent material obtained however has a much reduced density in comparison to the original compact superabsorbent, due to its expanded structure. This allows a great improvement in the control and homogeneity of distribution of superabsorbent particles in the final process of forming an absorbent structure. A further advantage of the present material is a relative reduction in cost per volume of the superabsorbent material.

The modification of the original compact structure of the superabsorbent material for obtaining an expanded structure in accordance with the invention is obtained by the expansion of the said original compact structure. The expanded superabsorbent polymer of the present invention has an absorption speed superior to that of the original compact polymer, principally in the initial period of absorption.

For example in the first five seconds, the expanded superabsorbent polymer shows absorption rates approximately 2 to 3.5 times higher than the original compact structured polymer.

The expansion may be performed by any suitable expansion process, which provides an expanded final structure. Preferably, thermoexpansion processes are used to perform the invention.

Among suitable thermoexpansion processes, the following are preferred:

a) expansion by submitting the compact superabsorbent material to thermotreatment in a microwave oven; and

b) expansion by contacting the compact superabsorbent material with an adequately heated surface.

The process of expansion which resulted in a highly satisfactory expanded structure was that of submitting the original superabsorbent material to thermotreatment in a microwave oven, and is thus the preferred expansion process of the invention.

5       The temperature of the thermotreatment may vary within a wide range from the softening temperature to a temperature lower than the carbonization temperature of the material. The preferred expansion temperature generally ranges from about 160 to 300°C more preferably from about 180 to 250°C.

10       Additionally, the expansion may be performed by a process which uses thermotreatment in association with a variation of pressure.

      This process, here referred as "gun type" expansion, consists in heating the superabsorbent compact material within a closed tube, until a pressure in the range of approximately 8 to 200 lb/in<sup>2</sup> is achieved, approximately 170 lb/in<sup>2</sup>. Thereafter the tube is opened so that the material contacts the atmosphere at a lower pressure, and the material is thereby expanded.

15       The expansion period is variable, and depends on the initial and final temperatures, the original moisture content of the polymer to prevent sticking of the polymer particles to each other and to the apparatus, the moisture being preferably between 0.5 and 5% and not greater than 15%. Generally, the preferred expansion time is between about 0.5 and about 30 minutes.

20       According to the present invention, any polymeric material based on sodium acrylate and having a compact structure may be used as a superabsorbent material to be expanded.

      The term "polymeric material based on sodium acrylate" as used here signifies a polymeric material constituted by monomeric units of acrylic acid (AA) and sodium acrylate (SA).

30       It has also been found according to the present invention that to obtain a satisfactory and efficacious expansion the proportion of AA and SA in the polymeric materials to be expanded should be in the range of 40 to 60% by weight of AA and 60 to 40% by weight of SA. Preferably, polymeric materials having from 50 to 60% by weight of SA are used. Polymeric material having a content of AA superior to these limits also permit expansion, but their particles become agglomerated during the heat treatment and thus impede the obtention of discrete expanded particles. A particularly preferred



superabsorbent material of the present invention is the superabsorbent polymer commercially known as Favor SAB, since the proportions of AA and SA monomers in the polymeric chain lies within the above ranges.

5 Any commercially available polymeric material based on sodium acrylate which does not have the cited proportions of AA to SA may also be expanded to provide the improved superabsorbent materials of the present invention, provided that they are duly treated so that the proportion of AA and SA monomers lies within  
10 the ranges specified above. This treatment consists essentially in transforming the AA groups present in the chain of the polymeric material into SA groups when the proportion of AA in the polymeric material is greater than 60% by weight, so that the quantitative ranges of AA and SA in the polymeric material to be expanded are  
15 within the limits established herein. This treatment, for example, may perform with any suitable compound for transforming SA groups into AA groups, for example by using an acidic compound such as hydrochloric acid or any suitable compound for transforming SA groups into SA groups, such as a basic sodium compound.

20 In the following list, intended to be illustrative only, the chemical composition of various types of commercially available superabsorbents based on sodium acrylate are given:

<u>Type of Material</u>	<u>Manufacturer</u>	<u>%AA</u>	<u>%SA</u>
Favor SAB	STOCKHAUSEN	50 - 57	50 - 43
25 Favor SAB 922	"	19 - 23	81 - 77
Aquakeep 10SH	KINTETSU	18 - 21	82 - 79
Arasorb 720	ARAKAWA	17 - 20	83 - 80
Dow XV 43.40800	DOW CHEMICAL	18 - 20	72 - 80
Arasorb 802	ARAKAWA	18 - 20	81 - 80
30 Arasorb 803	"	18 - 20	82 - 80
Arasorb 804	"	18 - 20	82 - 80

As may be readily seen in the above list, the only commercially available material having a proportion of AA and SA within the limits of the quantitative ranges of the present invention is  
35 Favor SAB. The other materials may be duly modified as already mentioned in order to obtain a polymeric material having proportions of AA and SA in the range of 60 to 40% by weight and 40 to 60% by weight respectively.

This commercial product is available in a granule size

ranging from 35 to 100 mesh, and may be readily expanded according to the present invention. However, it is preferable to use a particle size distribution between 50 and 100 mesh to provide greater uniformity and better absorption characteristics after expansion.

5       The superabsorbent expanded materials in accordance with present invention should have a final density in the range of 0.05 to 0.4 g/cm<sup>3</sup>, preferably, in the range of 0.05 to 0.14 g/cm<sup>3</sup>.

For illustrative purposes there are shown in the drawings the structures of absorbent materials of the prior art, and the  
10 structure of the new superabsorbent materials of the present invention, in which:

Figure 1 is a photomicrograph of the structure of natural sphagnum, magnified 675 times;

15       Figure 2 is a microphotograph of the structure of natural sphagnum magnified 675 times, showing the micropores;

Figure 3 is a microphotograph of the compact structure of a superabsorbent polymer (sodium polyacrylate) magnified 54 times;

20       Figure 4 is a microphotograph of a superabsorbent polymer (sodium polyacrylate) expanded according to the present invention and magnified 54 times;

Figure 5 is a microphotograph of the structure of an unexpanded superabsorbent polymer (sodium polyacrylate) magnified 270 times, and

25       Figure 6 is a microphotograph of a superabsorbent polymer (sodium polyacrylate) expanded according to the present invention and magnified 270 times.

30       The superabsorbent materials of the invention may be used either in isolation for the absorption of liquids, or in association with other materials to provide an absorbent structure.

The improved absorbent structures according to the invention include the new superabsorbent material in association with a suitable absorbent support. Such structures can comprise one or more layers or nucleus of said expanded superabsorbent material in  
35 association with one or more layers of an auxiliary material. Generally, wood pulp is used as the support. The quantity of superabsorbent material necessary to form an effective absorbent structure depends on the specific nature of the structure to be produced, and is well known to those skilled in this art. Proportions of about 2%

to 60%, preferably, 5% to 10% by weight of superabsorbent materials with respect to the weight of the support materials, preferably ground wood pulp, are satisfactory to provide such structure.

The absorbent structures above are suitable for forming absorbent articles such as disposable diapers, sanitary napkins, tampons and absorbent bandages or their like. The articles thus formed have effective liquid absorption and retention properties, without simultaneously presenting the disadvantages of conventional absorbent articles and the disadvantages presented by using the regular compact superabsorbent product without the expanded final structure.

#### Illustrative Examples

The superabsorbent starting polymer used in the illustrative examples is that commercially known by the name "Favor SAB", whose characteristics are listed below:

Chemical nature - Sodium polyacrylate

Appearance - white powder

Granin size - 160 to 500 microns

Apparent density -  $660 \pm 30$  g/l

Flowability - very good

Moisture content -  $5 \pm 2\%$

pH (bel at 1%) - 5.2 to 5.5

The expansion of the above material was performed in a microwave oven at 400 to 700 watts. The expansion time varied from about 0,5 to 30 minutes at a temperature of from 180 to 250°C.

In the following tables, some of the comparative results obtained in laboratory tests on compact regular Favor SAB and on the termally expanded Favor SAB, together with pulp and natural sphagnum, and absorbent structures formed from these materials.

Table II - Density Reduction

Grain Size Mesh	Bulk Density (g/cm <sup>3</sup> )		Expansion Parameters	
	Regular Favor SAB (not expanded)	Expanded Favor SAB	Temp. (°C) approx.	Time (Min approx.)
35 - 100	0,82	0,140-0,050	200	25
50 - 100	0,86	0,100-0,065	200	25
35 - 50	0,82	0,083-0,050	200	25
> 35				

Bulk Density determined through mass/volume relationship of 50 cm<sup>3</sup> of materials.

Expansion in lab scale through thermal treatment into a

Sanyo Microwave Oven - Model EM 90038, microwave frequency of 2450 MHz.

### Absorption Rate - Tea Bag Test

Test method: 0.2 g of superabsorbent material is set in heat sealable paper bags, commonly used commercially for tea bags. The dry weight is exactly determined and after the bags are sealed each sample is immersed in a 1% sodium chloride solution for intervals of 5, 10, 30, 60 and 120 seconds. After allowance to drain until excess solution is lost each weight is noted again. The same procedure as above described is repeated without superabsorbent.

The absorption after each immersion is calculated from the difference of both weight and converted to grams of saline solution absorbed per gram of material.

The results are listed in tests III.1, III.2 and III.3 below.

#### III.1 - Regular Favor SAB

Grain Size (mesh)	Absorption Rate (g/g)				
	After 5 sec	After 10 sec	After 30 sec	After 60 sec	After 120 sec
> 35	2,60	5,22	9,11	13,30	19,07
35 - 50	4,48	10,85	17,17	24,40	29,59
50 - 100	6,20	16,67	28,66	33,65	37,24

#### III.2 - Expanded Favor SAB Absorption Rate (g/g)

Grain size (mesh)	After 5 sec	After 10 sec	After 30 sec	After 60 sec	After 120 sec
> 35	9,15	13,93	19,02	24,85	30,66
35 - 50	11,71	17,67	23,99	29,76	33,78
50 - 100	12,95	21,48	30,17	34,36	38,59

#### III.3 - Regular and Expanded Favor SAB

	Absorption rate (g/g)	
	Regular Favor SAB	Expanded Favor SAB
After 5 seconds	2.60 - 6.20	9.15 - 12.95
After 10 seconds	5.22 - 15.67	13.93 - 21.48
After 30 seconds	9.11 - 28.66	19.02 - 30.17

	Regular Favor SAB	Expanded Favor SAB
After 60 seconds	13.30 - 33.65	24.85 - 34.36
5 After 120 seconds	19.07 - 37.20	30.66 - 38.59

The results above show an increasing of the initial absorption rate in the order of 2.0 to 3.5 times for the expanded Favor SAB in relation to the non expanded product.

#### 10 Liquid Retention

Cylindrical packs in form of tubes (to be introduced into centrifuge tubes) are manually prepared with light-weight non-woven material of about 20 g/m<sup>2</sup>.

15 Samples of 0,2 g of the superabsorbent materials were encased into them, the packs sealed and immersed in 1% sodium chloride solution. The samples were removed after two hours, allowed to drain for 10 minutes and were weighed and their respective total absorption rates were calculated (initial wet weight).

20 Thereafter the samples were subjected to two consecutive cycles of centrifuging, 15 minutes at 900 rpm and then 15 minutes at 1400 rpm.

The quantity of liquid retained by the sample was calculated by difference between the weights registered before and after the centrifuging cycles and were expressed in percentage terms.

25 The results are shown in table IV below:

Table IV - Liquid Retention

		After centrifuging at				
		900 rpm			1400 rpm	
30		Initial Absorption (%)	Residual Absorption (%)	Retention (%)	Residual Absorption (%)	Retention (%)
	Ground wood pulp	1760	82	4.6	64.5	3.6
	Sphagnum	1865	569	30	379	20
35	Regular Favor SAB	3180	2280	71.7	2065	64.9
	Expanded Favor SAB	3074	2125	69.1	1860	60.5

The results above show a practically unaffected retention capacity in the expanded Favor SAB in relation to the Regular

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Favor SAB.

Reduction of Blocking effectTest Method

Sample disks of ground wood pulp and ground wood pulp  
 5 containing regular and expanded Favor SAB were manually prepared  
 in lab scale. They were mixed through a kitchen blender, vacuum  
 compacted in a 9 cm diameter mould and pressed to a thickness be-  
 tween 1.0 and 1.5 mm.

Individual sample disks weighed approximately 2.0 g.

10 A 2 x 2 cm pad of a wood pulp structure with an infe-  
 rior skin of PCAc was centrally disposed on the disks.

Five ml of a 1% sodium chloride solution was added to  
 this 2 x 2 cm pad to be absorbed by the disks.

After 2 minutes the stained area of the disk and the a-  
 15 mount of saline solution absorbed by the disk were determined.

The area was measured by a planimeter and the quantity  
 of absorbed solution was determined from the difference of final  
 and initial weights of the disks.

Table V - Reduction of blocking effects

20 Disk Composition (%)

	Ground wood pulp	Regular FAVOR SAB	Expanded FAVOR SAB	Stained area (cm <sup>2</sup> ) On disk	Liquid Absorbed
	100	-	-	37.1	73.4
25	90	10	-	35.2	72.4
	90	-	10	35.9	77.4
	70	30	-	21.2	67.8
	70	-	30	27.6	74.8
30	40	60	-	13.4	59
	40	-	60	22.0	55.4

Notes 1. A larger stained area indicates a reduced blocking action.

2. More liquid absorbed indicates as higher ability to  
 "pump" liquids.

35 The detail of Table V above shows the superior perfor-  
 mance of expanded Favor SAB.

Absorption capacity and velocity.Test Method

GATS apparatus, 1% NaCl saline solution, pressure of

0.05 psi and orifice centralized in the plate.

Disks manually prepared in the laboratory, mixed in liquidizer, vacuum compacted, in a 9 cm diameter mould and pressed at 1 ton pressure. Pads weighed between 1.856 and 1.970 g.

5 Table VI - Absorption capacity and velocity for experimental absorbent structures.

Experimental structure	Pads density (g/cm <sup>3</sup> )	Absorption capacity (g/g)	Absorption velocity (g/min)
10 100% ground wood pulp	0.123 - 0.142	8.2 - 7.5	11.4 - 11.9
90% pulp + 10% expanded Favor SAB	0.133	12.1	3.24
15 90% pulp + 10% expanded Favor SAB	0.139 - 0.147	11.1 - 11.3	8 - 8.8

20 The details above show an increase in absorption velocity of the structure of the invention by about 2.5 times relative to the structure using normal Favor SAB (3.24g/min as opposed to  $\pm$  8.4 g/min).

25 The improvements listed above are of great importance, since the superabsorbents of the present invention present, in combination, a high velocity of absorption, a high capacity for absorption, a satisfactory liquid retention and low density due to the expansion. This is not presented by any of the conventional absorbent materials cited here.

30 Various other comparative test results will be shown in the following tables, comparing samples of absorbent structures prepared with the compact superabsorbent and with the expanded superabsorbents of the present invention.

#### Experimental absorbent structures

35 Experimental absorbent structures as similar as possible to commercial product were prepared in lab scale.

They were formed by the deposition of two layers of ground wood pulp, the inferior one having a PCAC skin, aiming to reproduce as much as possible an existing commercial sanitary pad. The total weight of this structure was in the order of 6,0 g (3,0

g per layer) and with dimensions of about 18,0 x 7,0 cm (around 126 cm<sup>2</sup>).

The final absorbent structure was composed with the superabsorbent materials spread between the two layers of ground wood pulp.

Samples:

A - Control - 100% ground wood pulp around 6,0 g final weight and around 126 cm<sup>2</sup>.

B - Composite containing 2% of expanded Favor SAB spread between the two layers of ground wood pulp.

C - Composite containing 10% of expanded Favor SAB spread between the two layers of ground wood pulp.

D - Composite containing 10% of the Regular Favor SAB (not expanded) spread between the two layers of ground wood pulp.

Test Method:

GATS apparatus, pressure of 0,05 psi, 1% sodium chloride solution, point source.

The results are listed in the table VII below.

Table VII - Absorption characteristics at 0.05 psi

Composite structure	Control (100% pulp) (A)	Expanded Favor SAB		Regular Favor SAB (D)
		2% (B)	10% (C)	
Density (g/cm <sup>3</sup> )	0.114	0.113	0.116	0.125
Initial volume (cm <sup>3</sup> )	11.92	12.02	12.49	11.98
Final volume (cm <sup>3</sup> )	9.96	10.60	12.69	13.57
Volume variation (%)	-16.5	-11.7	+ 1.6	+13.3
Absorption capacity (g/g)	9.6	10.4	11.9	12.8
Absorption velocity (g/min)	22.9	22.7	14.4	7.1

As shown by the above table, the advantages of the expanded superabsorbent lie in the results of volume variation and absorption velocity when compared to structures containing the same amount of superabsorbent, one being unexpanded the other being ex-



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panded.

### Fluid Distribution

#### Test Method

45 ml of 1% sodium chloride solution is discharged  
5 through a burette in the central point of an experimental absor-  
bent structure similar to the one described for Table VII.

The solution was poured in 3 consecutive discharges of  
15 ml each, at intervals of 30 minutes.

The wetted absorbent structure is then cut in 6 equal  
10 sections ( $S_1$ - $S_6$ ) and the quantity is determined as well as the  
percentage of utilization of the absorbent structure.

Illustrative sections of the structure:

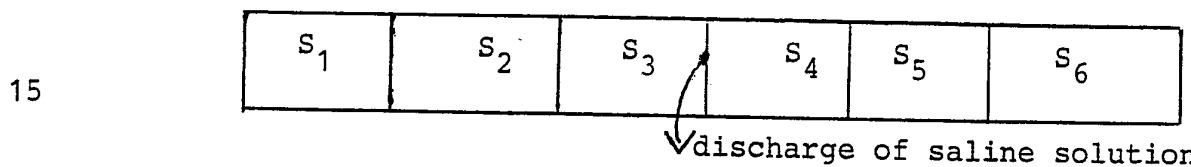


Table VIII below indicates improved results of distri-  
bution and utilization in the structures containing the expanded  
Favor SAB over those ones containing the unexpanded regular Favor  
20 SAB.

Table VIII - Fluid Distribution and percentage of utilization  
Absorption of each section (g/g)

25	Sections	Control (100% Pulp)	Expanded	Favor SAB	Regular Favor SAB
			2%	10%	
30	$S_1$	7.1	7.2	6.4	5.4
	$S_2$	7.9	8.3	7.6	7.3
	$S_3$	7.9	8.3	8.8	8.4
	$S_4$	8.2	8.4	8.4	9.1
	$S_5$	8.2	8.6	8.0	7.7
	$S_6$	9.5	7.6	6.1	6.5
35	Utilization (%)	93	93.5	88.1	82.8

The % of utilization is calculated as follows:

$$\% \text{ of utilization} = \frac{\text{Average Wetness} - \text{Absorb. Average Deviation}}{\text{Average Wetness}} \times 10$$

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Pumping PowerTest Method

A 2 x 2 cm pad of ground wood pulp with PCAc skin was centrally disposed on the absorbent structure similar to that described for Table VII.

30 ml of 1% saline solution is poured in the 2 x 2 pads in 6 consecutive discharges of 5 ml each at intervals of 10 minutes.

The pumping power of the absorbent structure is then measured by weighing the quantity of solution each structure pumped from the 2 x 2 pad after each discharge.

The results according to Table IX below indicate the higher pumping effect of the structures containing the expanded Favor SAB structure containing the regular Favor SAB, principally at the initial discharge of the saline solution.

Table IX - Pumping Power

	Fluid added (cm <sup>3</sup> )	Control 100% pulp	Expanded Favor SAB		Regular Favor SAB
			2%	10%	
		A	B	C	D
	5	12.6	16.4	19.7	5.2
	10	35.8	44.9	45.1	30.2
25	15	58.3	62.3	62.7	52.6
	20	65.6	68.6	69.7	62.2
	25	70.4	72.3	72.8	68.2
	30	75.4	75.4	75.8	72.4

Example "X"

This example illustrates the chemical modification of a commercially available sodium acrylate based polymer to obtain a proportion of AA and SA monomers within the operative ranges according to the present invention.

As starting materials, ARASORB 720, FAVOR SAB 922 and DOW XV 43 48800 having 80% of SA and 20% of AA were used. Each of these materials was treated with hydrochloric acid to obtain a polymeric material having 50% by weight of SA and 50% by weight of AA. There were used potentially 0.32 eqg of HCl for each 100g of starting material to perform the substitution of 0.32 eqg of

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positive sodium ions for each 100g of starting material, and thus obtain a polymeric material having 50% AA and 50% SA. When submitted to thermo expansion of Favor SAB in the first example, there were obtained polymeric materials having an expanded structure analogous to the expanded structure obtained starting with Favor SAB.

It should be clear that the examples above are indicated here only to illustrate the present invention; and thus should not be limitative thereof in any way. The scope of the invention is clearly much broader than that described in the specific examples presented here.

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## Claims:

1. A superabsorbent material having improved properties, characterized by the fact of consisting of a superabsorbent polymeric material based on sodium acrylate, whose original structure  
5 was modified by expansion so that the resultant final structure is an expanded structure of low density.

2. A superabsorbent material in accordance with claim 1, characterized by the fact that the said superabsorbent polymeric material based on sodium acrylate is constituted by monomers of  
10 acrylic acid and sodium acrylate.

3. A superabsorbent material in accordance with claim 1 or claim 2, characterized by the fact that the said superabsorbent polymeric material based on sodium acrylate to be expanded comprises from 60 to 40% by weight of sodium acrylate and 40 to 60% by  
15 weight of acrylic acid.

4. A superabsorbent material in accordance with claim 1 or claim 2, characterized by the fact that the sodium acrylate based polymeric material comprises preferably 50 to 60% by weight of acrylic acid and 50 to 40% by weight of sodium acrylate.

20 5. A superabsorbent material in accordance with claim 1, characterized by the fact that the granule size of the original superabsorbent polymer is between 35 to 100 mesh.

6. A superabsorbent material in accordance with claim 1, characterized by the fact that the density of the expanded super-  
25 absorbent polymer is from approximately 0.05 to approximately 0.4g/cm<sup>3</sup>, preferably, 0.05 to 0.14 g/cm<sup>3</sup>.

7. An absorbent structure having improved properties comprising a superabsorbent material and a suitable support, characterized by the fact that the said superabsorbent material is a super-  
30 absorbent polymeric material based on sodium acrylate, whose original structure was modified by expansion so that the resultant super absorbent particles have an expanded structure.

8. An absorbent structure in accordance with claim 7, characterized by the fact that the polymeric material based on  
35 sodium acrylate is constituted of monomers of acrylic acid and sodium acrylate.

9. An absorbent structure in accordance with claim 7 or claim 8, characterized by the fact that the polymeric material based on sodium acrylate comprises 60 to 40% by weight of sodium

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acrylate and 40 to 60% by weight of acrylate acid.

10. An absorbent structure in accordance with claim 9, characterized by the fact that the polymeric material based on sodium acrylate comprises preferably 50 to 60% by weight of acrylic acid and 40 to 50% by weight of sodium acrylate.

11. An absorbent structure in accordance with claim 7, characterized by the fact that the said support is wood pulp.

12. An absorbent structure in accordance with claim 6, characterized by comprising one or more layers or nucleous the said expanded superabsorbent polymer in association with one or more layers of an auxiliary absorbent material.

13. An absorbent article, by comprising an absorbent structure as defined in claim 7.

14. An absorbent article in accordance with claim 13, characterized in that it is selected from the group comprising disposable diapers, sanitary napkins, tampons, and absorbent bandages.

15. Process for preparing a superabsorbent material with improved properties, characterized by the fact of comprising thermo treatment of a superabsorbent polymeric material based on sodium acrylate and having a compact structure, so that the compact structure is expanded to provide a final polymeric material of low density structure.

16. Process in accordance with claim 15, characterized by the fact that the superabsorbent polymeric material based on sodium acrylate is constituted by monomers of acrylic acid and sodium acrylate.

17. Process in accordance with claim 15 or claim 16, characterized by the fact that the polymeric material based on sodium acrylate comprises 60 to 40% by weight of sodium acrylate and 40 to 60% by weight of acrylate acid.

18. Process in accordance with claim 17, characterized by the fact that the said polymeric material based on sodium acrylate comprises preferably 50 to 60% by weight of acrylic acid and 40 to 50% of sodium acrylate.

19. Process in accordance with claim 15, characterized by the fact that said thermo treatment is performed at a temperature of from the softening temperature up to a temperature lower than the temperature of carbonization of the said compact structure

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polymer.

20. Process in accordance with claim 19, characterized by the fact that the said temperature is preferably in the range of about 180 to 250°C.

5           21. Process in accordance with claim 15, characterized by the fact that the said compact structure polymer has a particle size of between 35 to 100 mesh.

22. Process in accordance with claim 21, characterized by the fact that the particle size is from 50 to 100 mesh.

10           23. Process in accordance with claim 15, characterized by the fact that the moisture content of the compact structure polymer to be expanded is not greater than 15%.

24. Process in accordance with claim 15, characterized by the fact that the moisture content is between 0.5 to 5%.

FIG. 1



FIG. 2





FIG. 3

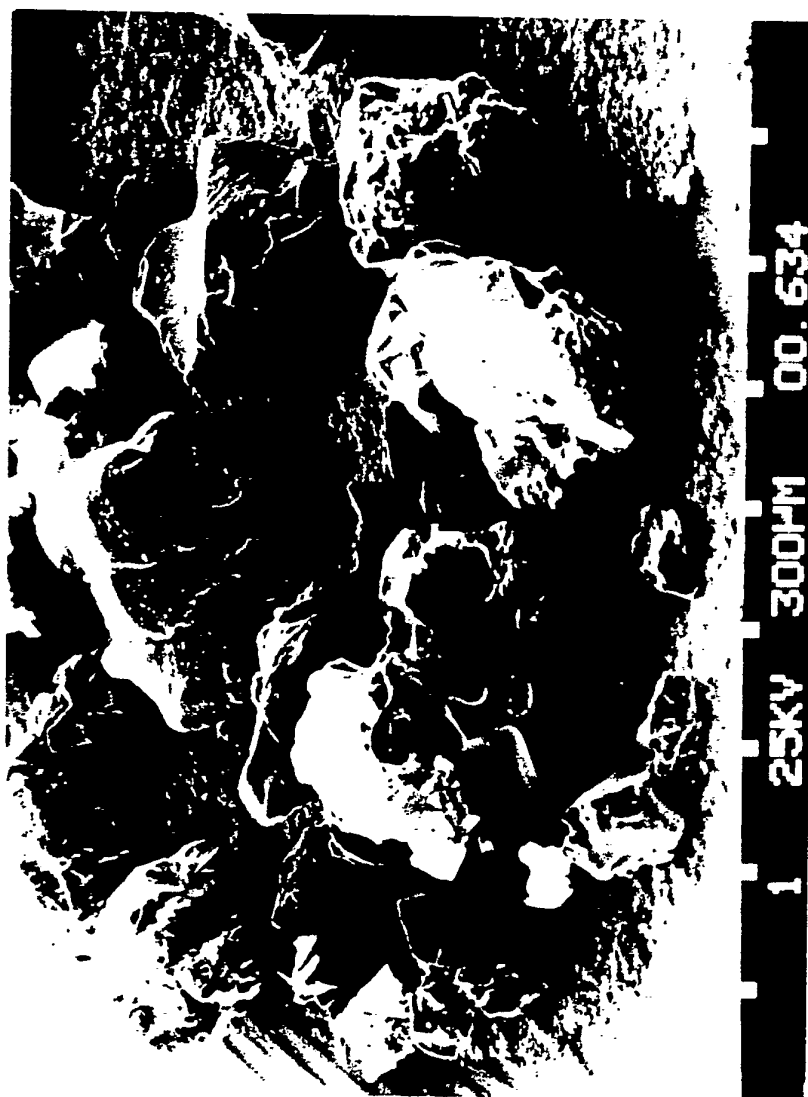


FIG. 4

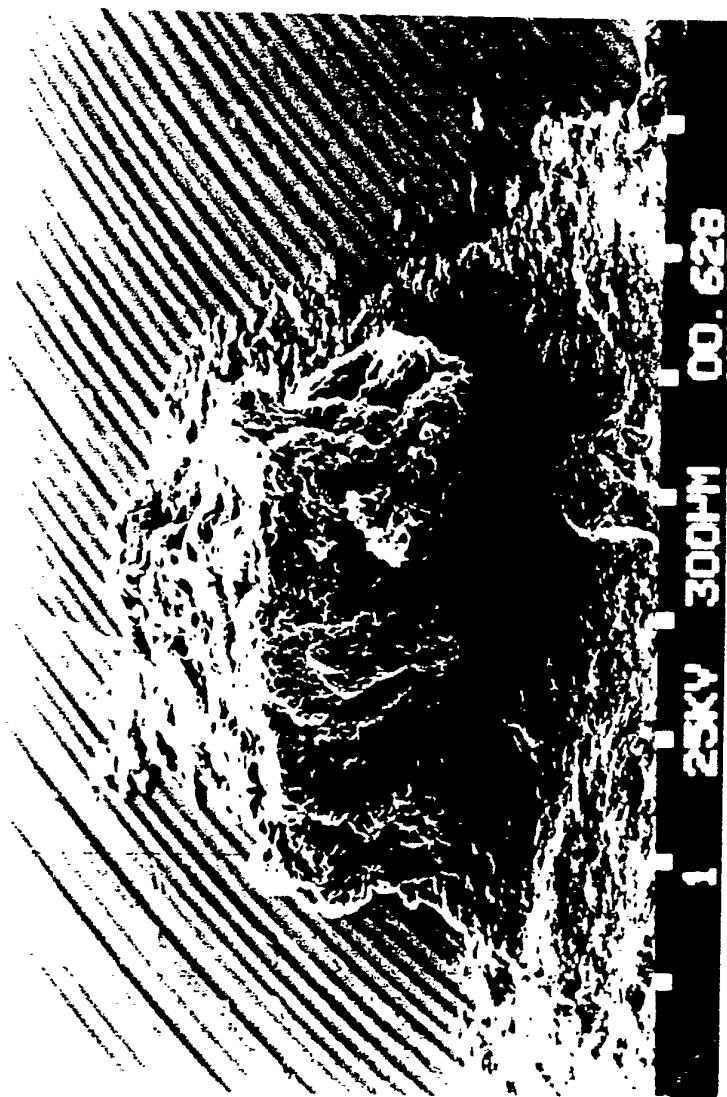


FIG. 5

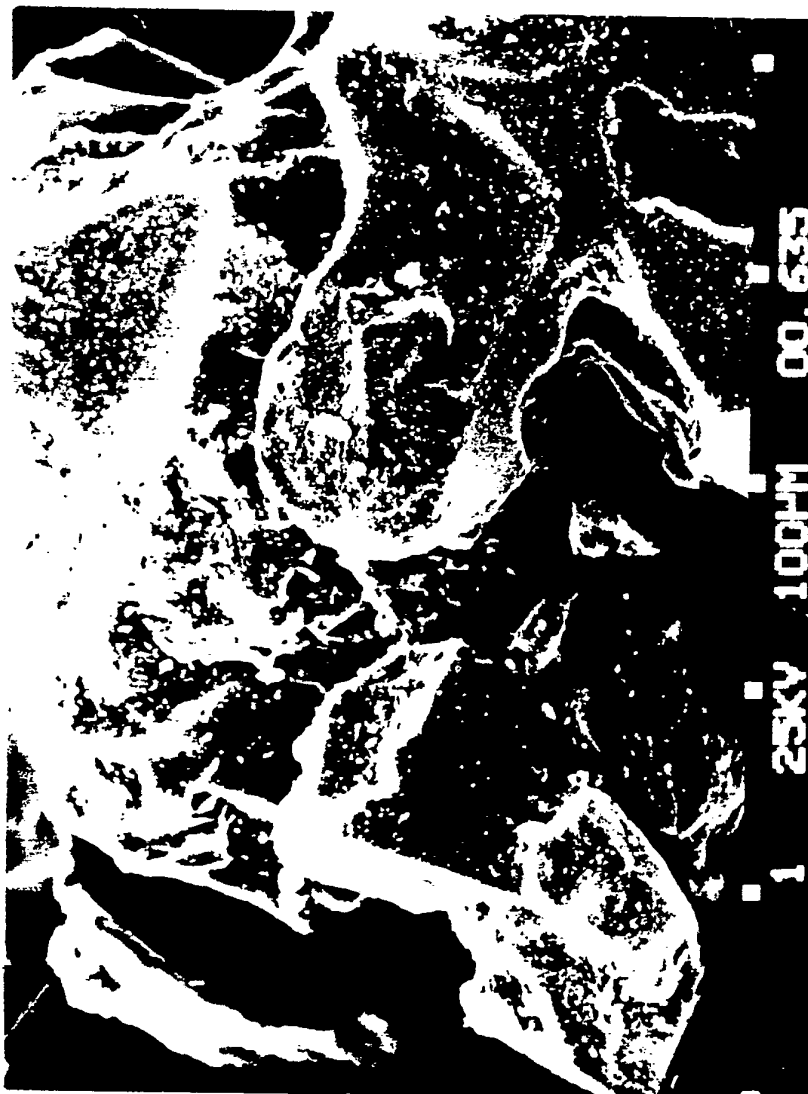
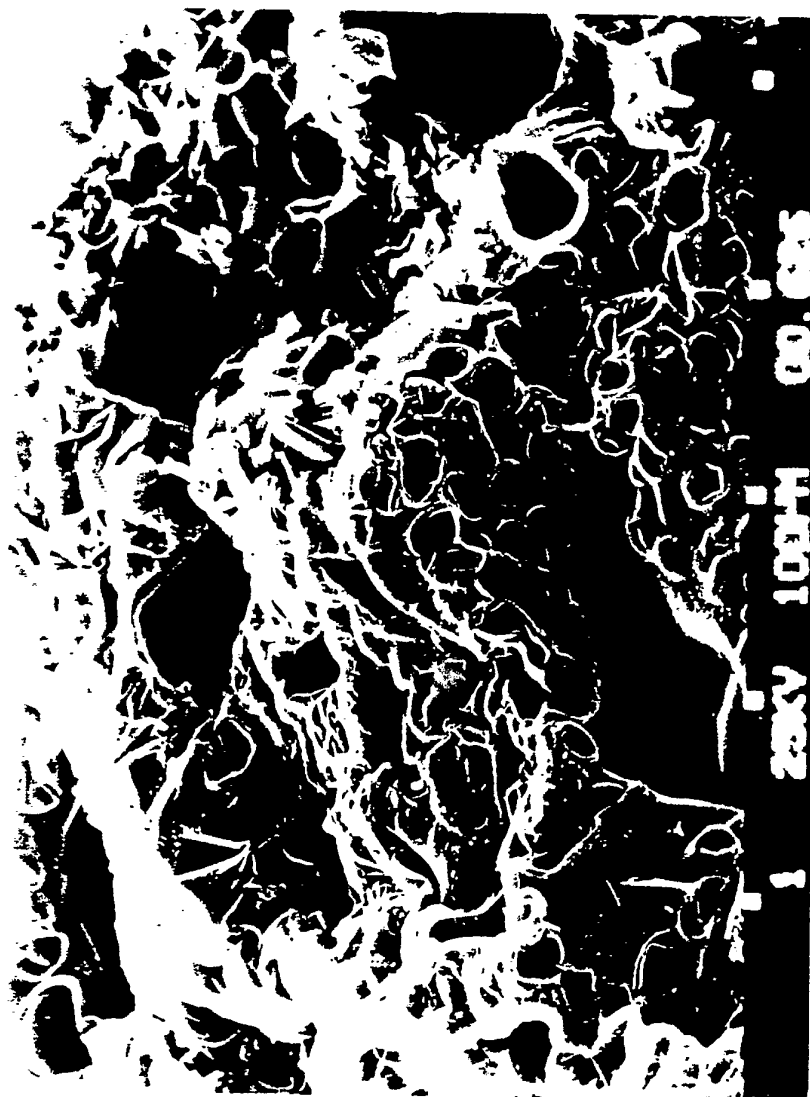


FIG. 6



# INTERNATIONAL SEARCH REPORT

International Application No PCT/BR 90/00009

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (if several classification symbols apply, indicate all) <sup>6</sup>		
According to International Patent Classification (IPC) or to both National Classification and IPC <b>IPC5: A 61 L 15/24, 15/60</b>		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched <sup>7</sup>		
Classification System	Classification Symbols	
IPC5	A 61 L; A 61 F	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in Fields Searched <sup>8</sup>		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT<sup>9</sup></b>		
Category *	Citation of Document, <sup>11</sup> with indication, where appropriate, of the relevant passages <sup>12</sup>	Relevant to Claim No. <sup>13</sup>
X	EP, A2, 0295438 (JOHNSON & JOHNSON PATIENT CARE, INC.) 21 December 1988, see abstract, examples 2,5 and claims  --	1,15
X	EP, A1, 0049944 (JOHNSON & JOHNSON) 21 April 1982, see pages 3,5; page 12, line 11 - page 13, line 4, claims  --	1-2
Y	US, A, 4902721 (PHAM ET AL) 20 February 1990, see the whole document  --	1-24
<div style="display: flex; justify-content: space-between;"> <div style="width: 48%;"> <p><b>* Special categories of cited documents:<sup>10</sup></b></p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"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)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 48%;"> <p>"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</p> <p>"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"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.</p> <p>"&amp;" document member of the same patent family</p> </div> </div>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
22nd November 1990	04.12.90	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	R.J. Eernisse	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No
Y	EP, A2, 0347241 (MITSUBISHI PETROCHEMICAL CO., LTD.) 20 December 1989, see abstract, page 3, line 40 - line 57, example 3, claims  -- -----	1-24

**ANNEX TO THE INTERNATIONAL SEARCH REPORT  
ON INTERNATIONAL PATENT APPLICATION NO. PCT/BR 90/00009**

SA 39941

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.  
The members are as contained in the European Patent Office EDP file on 01/11/90  
The European Patent office is in no way liable for these particulars which are merely given for the purpose of information.

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A2- 0295438	21/12/88	AU-D- 1608188	17/11/88
		JP-A- 63302941	09/12/88
		US-A- 4808637	28/02/89
EP-A1- 0049944	21/04/82	AT-E- 10257	15/11/84
		AU-B- 548161	28/11/85
		AU-D- 7445581	04/03/82
		CA-A- 1183513	05/03/85
		GB-A-B- 2083487	24/03/82
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		JP-A- 57073051	07/05/82
		OA-A- 6890	30/04/83
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		US-A- 4394930	26/07/83
		US-A- 4415388	15/11/83
US-A- 4902721	20/02/90	US-A- 4853420	01/08/89
		US-A- 4885315	05/12/89
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		JP-A- 2034607	05/02/90

For more details about this annex : see Official Journal of the European patent Office, No. 12/82