PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 5 : **B01J 13/04, 13/20, 13/22** **A23L 1/216, 1/236**	**A1**	**(11) International Publication Number:** **WO 92/11084** **(43) International Publication Date:** 9 July 1992 (09.07.92)

(21) International Application Number: PCT/US91/09434

(22) International Filing Date: 17 December 1991 (17.12.91)

(30) Priority data:
629,721 17 December 1990 (17.12.90) US

(60) Parent Application or Grant
(63) Related by Continuation
US 629,721 (CIP)
Filed on 17 December 1990 (17.12.90)

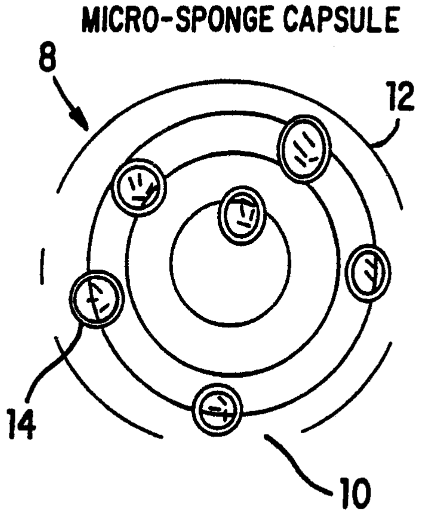
(71)(72) Applicants and Inventors: REDDING, Bruce, K., Jr. [US/US]; 2708 S. 86th Street, Philadelphia, PA 19153 (US). BUTCHER, Brian [US/US]; Park Boulevard, Apartment D, Cherry Hill, NJ 08034 (US). GARRISON, Walter, S. [US/US]; 715 Auburn Road, Fairless Hills, PA 19030 (US). SCHMUCKER, Arden, E. [US/US]; 7922 Allen Drive, Alliance, OH 44601 (US).

(74) Agent: LIPTON, Robert, S.; Lipton & Famiglio, P.O. Box 546, Media, PA 19063 (US).

(81) Designated States: AT (European patent), AU, BE (European patent), BR, CA, CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, KR, LU (European patent), MC (European patent), NL (European patent), SE (European patent), SU+, US.

Published
With international search report.

(54) Title: MICROENCAPSULATED SWEETENERS FOR USE IN BAKED GOODS



(57) Abstract

Natural and artificial sweeteners, including Aspartame, are encapsulated in a protective shell material (4) which will enable the sweetener (6) to resist the temperature, moisture and Ph effects presented during the baking process of cookies, pies, cakes, crackers, puddings, microwave treated foods an any other food to which the sensitive natural or artificial sweetener is to be added which will then be subjected to heating. The encapsulation process and materials mentioned in this application enable the survival of the sweeteners' sweetness through the baking cycle.

+ See back of page

+ DESIGNATIONS OF "SU"

Any designation of "SU" has effect in the Russian Federation. It is not yet known whether any such designation has effect in other States of the former Soviet Union.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria
AU	Australia
BB	Barbados
BE	Belgium
BF	Burkina Faso
BG	Bulgaria
BJ	Benin
BR	Brazil
CA	Canada
CF	Central African Republic
CG	Congo
CH	Switzerland
CI	Côte d'Ivoire
CM	Cameroon
CS	Czechoslovakia
DE	Germany
DK	Denmark
ES	Spain
FI	Finland
FR	France
GA	Gabon
GB	United Kingdom
GN	Guinea
GR	Greece
HU	Hungary
IT	Italy
JP	Japan
KP	Democratic People's Republic of Korea
KR	Republic of Korea
LI	Liechtenstein
LK	Sri Lanka
LU	Luxembourg
MC	Monaco
MG	Madagascar
ML	Mali
MN	Mongolia
MR	Mauritania
MW	Malawi
NL	Netherlands
NO	Norway
PL	Poland
RO	Romania
SD	Sudan
SE	Sweden
SN	Senegal
SU+	Soviet Union
TD	Chad
TG	Togo
US	United States of America

MICROENCAPSULATED SWEETENERS FOR USE IN BAKED GOODS

Technical Field

This invention relates to the use of encapsulation to provide stability of artificial and certain natural sweeteners in baked good applications such as cakes, pies, cookies and certain confectionery products including candies. More specifically, this invention relates to the encapsulation of Aspartame to provide for small capsules with Ph, temperature and moisture stability.

Background Art

Aspartame is an artificial sweetener currently in use in "cold" food applications and short term heated applications. Examples of cold applications include soda, toothpaste, and snack foods processed at room temperature. Short term heated applications include coffee and tea beverages. Aspartame has not heretofore been effective in baked goods where a combination of Ph, high temperatures and moisture content may act to chemically degrade the Aspartame. Items such as cakes, cookies and pies which may be baked at elevated temperatures for several minutes to nearly an hour tend not to be good candidates for use by certain temperature, Ph, or moisture sensitive sweeteners including Aspartame.

Several efforts have been attempted to produce a bake survivable Aspartame product. Modifications to the compound have not been effective. Coating the Aspartame powder to protect it from the ravages of prolonged high temperature exposure, Ph and moisture effects seems a viable approach but conventional encapsulation methods have led to microencapsulated Aspartame products which suffer several problems:

1. The capsule sizes produced were so large as to affect the distribution of the capsules throughout the batter. Large capsules do not distribute well, resulting in many areas of a cake or cookie which were void of sweetness or which were aesthetically undesirable due to a speckled appearance in the product.

2. In the prior art, fat/wax materials were used as capsule

shells for Aspartame. Under observation the capsules were found to suffer from a series of cracks and lesions. This is thought to be caused by the wax as it hardens from a molten state into a solid form. High Ph water, such as produced in many cake formulations, may then enter the shell through these cracks and directly affect the core of Aspartame.

3. Single, thin shelled, microcapsules are far too weak to withstand the shear of stirring applications,, and often break when placed in conventional food or cake batter mixing devices.

4. The use of wax and other coatings often leads to a static charge on the capsule shell, leading to agglomeration of the capsules. This prevents adequate dispersion of the capsules throughout the batter, again leading to void spots.

The invention described herein is a means of avoiding these problems. The current invention provides for smaller capsules, with little agglomeration, and with a more complete enclosure of the capsule shell, thereby preventing or reducing the number of cracks or fissures which have led to past problems.

Brief Description of the Figures

Figure 1 depicts several microcapsule constructions available to encapsulate sweeteners.

Figure 2 is a DSC thermographic spectrum of granulated Aspartame using a fat/wax binding material.

Figure 3 is a DSC thermographic spectrum of the encapsulated Aspartame using a natural polymer as the shell coating of the capsule.

Figure 4 depicts the decomposition products produced by Aspartame when subjected to the temperatures, Ph and moisture present during most baking operations.

Figure 5 is a graph illustrating the decomposition ranges of Aspartame at various Ph levels at a standard temperature.

Figure 6 depicts the use of fats or waxes in the encapsulation of

Aspartame, illustrating the use of normal fat/wax coatings vs. a tempered fat/wax coating.

Figure 7 depicts a block diagram of the process used to temper fats and waxes as applied in the encapsulation of particulate sweeteners.

Figure 8 depicts a prior art method of encapsulation known as coacervation.

Detailed Description of the Invention

Food sweeteners, which may be either natural, artificial or a combination of both natural and artificial materials, are coated with a substance which will enable the sweeteners to survive the effects of and the conditions present in various baked goods, and additionally to provide a means of reducing any aftertaste associated with the use of certain artificial sweeteners. A partial list of natural sweeteners and artificial sweeteners are shown, respectively in Tables 2A and 2B.

Reference is made to U..S. Patent No. 4,384,004 by T. Cea, et al. and U.S. Patent No. 4,704,288 by J. Tsau, et al. Both patents describe encapsulated Aspartame. Tsau describes the use of encapsulated Aspartame in the field of baked good applications. Tsau teaches coatings comprised of fat materials, and capsule sizes over 177 microns in diameter. It has been found that these capsules produce void spots in various baked goods including cakes, pies and cookies. "Void Spots" are defined as areas within the baked good which are not sweet, being either totally devoid of sweeteners, bland to the taste, or actually abhorrent to the taste.

Void spots may occur because the capsules are not completely formed, thereby offering little protection for the Aspartame leading to degradation of the core material during the baking process (Aspartame is known to be Ph, moisture and temperature sensitive, resulting in a breakdown of the Aspartame molecule into certain distasteful or bland decomposition by products, as described in Figure 4).

The void spots may also form because the capsules are too large thereby preventing adequate dispersion through the batter. Void spots result in poor distribution within the baked good leaving void spots in many sections which are then devoid of taste. Table 5 illustrates the survivability of sweetness after baking chocolate cake with regular Aspartame sweetener when used in either raw or granulated form as compared to various samples of encapsulated Aspartame. Note that the granulated Aspartame, which is shown to have an average particle size over 650 microns according to the size comparison as shown in Table 6, is found to leave several void spots according to the taste tests conducted and presented in Table 3.

Void spots also cause a speckled appearance of the cake as the large particles survive baking completely intact and are clearly visible especially on dark colored baked products such as chocolate cake. Table 1 shows the appearance of both raw and granulated Aspartame in cake in relation to encapsulated Aspartame samples of a smaller average size. The raw Aspartame has an average particle size of just 29 microns, as shown in Table #6 while the granulated Aspartame has an average size over 650 microns. The leaves specks in the baked good which are especially visible on the underside of cakes, pies and cupcakes as evidenced by the descriptions present in Table 1.

TABLE #1

CAKE APPEARANCE USING ENCAPSULATED ASPARTAME
VS.
RAW AND GRANULATED ASPARTAME

	SPECKLED APPEARANCE
Raw Aspartame	5-7
Granulated Aspartame	0-1
AES-B-36	10
AES-B-39	10
AES-B-40	10
AES-B-43.1	10

TABLE #1 (CONT'D)

SCALE: Heavily speckled appearance (0) to non-visible specs (10)

10 = No specs visible on underside or throughout cake

5 = Some specs visible

0-1 = Heavily speckled cake, very poor aesthetic appearance

TABLE #2A

PARTIAL LIST OF NATURAL SWEETENERS

PRODUCT	SUPPLIER	COMPOSITION
Sugar	Imperial Sugar Co.	Sucrose
Lactose	Plalte & Banner, Inc.	Milk Sugar
Corn Syrup	Corn Products, Co.	Corn Sugar
Corn Syrup Solids	Corn Products, Co.	Corn Sugar
Dextrose	Corn Products, Co.	Corn Sugar
Fructose	Corn Products, Co.	Fruit Sugar
Mannitol	Corn Products, Co.	C6 H14 06

TABLE #2B

PARTIAL LIST OF ARTIFICIAL SWEETENERS

PRODUCT	SUPPLIER	COMPOSITION
Nutrasweet	Nutrasweet	Aspartame
ACE-K (Sunett)	Hoechst-Celenese	Acesulfame K
Cyclamate	Abbott Labs	Salts of Cyclamic Acid
Suralose	McNeil	Chlorinated Derivative of Sucrose
Alitame	Pfizer	Amino Acid Based
Saccharin	PMC Specialities	Derived From Methyl Anthranilate
Left Handed Sugars	Biosphere	

Additionally, the void spots may occur because the large granulated sweetener may not completely release the sweetener during the baking process or immediately afterwards, thereby not allowing contact between the user and the sweetener's active material at the taste point. The taster then perceives no sweet taste in the baked good at all. Additionally, these large capsules provide a severe aftertaste problem for many users after the baking process.

To correct the problems of void spots and speckling in various baked goods, alternative formulations using smaller capsules must be employed. Smaller capsules provide for greater dispersion in a homogeneous manner within the batter, resulting in cakes, cookies and pies with no visible sweetener specks, and with fewer or no void spots. Whereas the prior art teaches the use of an Aspartame capsule, which is really a granulated Aspartame particle, with a minimum size of 177 microns and an average size over 400 microns, the present invention produces capsules with an average size as low as 4.0 microns. Cakes, cookies and pies produced with these sweetener capsules exhibit no void spots at all and no visible specks in the final product after baking. Note the results indicated in the size comparison shown on Table #6, the sweetness taste test illustrated in Table 3 and the aesthetic appearance comparison in chocolate cake as shown in Table 1.

TABLE #3A

COMPARISON OF SWEETNESS DISTRIBUTION IN CHOCOLATE CAKE LOADED AT 1.25 GM ASPARTAME EQUIVALENT ACTIVE IN CAKE MIX

	SWEETENER DISTRIBUTION
Raw Aspartame	1
Granulated Aspartame	3
AES-B-36	10
AES-B-39	10

TABLE #3A (CONT'D)

	SWEETENER DISTRIBUTION
AES-B-40	10
AES-B-43.1	10

SCALE: Worst (O) to Best (10)

10 = Sweet in every portion of cake, no void spots detected.

5 = Some void spots, moderate sweetener.

0-1 = Barely any detectible sweetness.

Results based on a subjective blind taste test conducted using 20 persons.

TABLE #3B

COMPARISON OF AFTER TASTE PERCEPTION IN CHOCOLATE CAKE LOADED AT 1.25 gm ASPARTAME EQUIVALENT ACTIVE IN CAKE MIX

	AFTERTASTE PERCEPTION
Raw Aspartame	10
Granulated Aspartame	8
AES-B-36	2
AES-B-39	3
AES-B-40	2
AES-B-43.1	1

SCALE: Least amount of aftertase (O)
Most amount of aftertaste (10)

10 = High presence of after taste.

TABLE #3B (CONT'D)

5 = Moderate after taste.

1 = Barely any perceived after taste.

Results are based on a subjective blind taste test conducted using 20 persons.

Microscopic examination of capsules made by prior art methods shows that the fat/wax material used often has severe cracks and lesions in the surface, and that these cracks become more pronounced as the capsules age.

The capsules of the prior art, therefore, offer little protection against the degradation effects of Ph, moisture and temperature presented during the preparation of the dough or during the baking of various pies, cakes, cookies and other baked goods. The fat/wax shell material is not an effective barrier due to the micropores and crevices in the structure as illustrated in Figure 6.

While it has been discovered by applicants that an improved fat/wax material in all beta or nearly all beta crystalline form provides a more protective coating in the encapsulation of APM (Aspartame), other materials have also been observed to provide similar protective abilities. These other materials include natural polymers such as those listed in Table 4.

TABLE #4

MATERIALS AVAILABLE FOR CAPSULE SHELLS IN ENCAPSULATED SWEETENERS

NATURAL POLYMERS

GUM ARABIC
ETHYLCELLULOSE
MODIFIED CORN STARCH
GLYCEROL TRISTEARIN
METHYLCELLULOSE
CELLULOSE ACETATE PHTHALATE
PROPYLHYDROXYLCELLULOSE
GLYCEROL TRISTEARIN
CARNOLA WAX
SOYA FLAKES
RAPESEED WAX
CARNAUBA WAX
MICROWAX (PETROLEUM)
SPECIAL FAT 42,44, 168 T
BE SQUARE WAX 195W
ASTOR WAX 180

PROTEINS
SUCCINYLATED GELATIN
NITROCELLULOSE
GELATIN
MODIFIED STARCHES
ARABINOGELACTAN
STEROTEX HM, K
COTTON FLAKES
CASTOR WAX
BEESWAX
CANDELITTA WAX
BOLAR WAX 1014
BE SQUARE WAX 195A
ENERGYBOOSTER
ASTOR WAX 150

The encapsulation of Aspartame using a fat/wax based coating is possible using a fat which has been forced into a more stable beta crystalline form by heat treatment methods employing either a thermal tempering of the fat while in a molten state or soon after it has solidified to help prevent the formation of lesions and micropores in the coated sweetener product. Additionally, fats and waxes may be melted into a molten liquid state which is then subjected to an abrupt pressure change before the molten fat or wax is applied as a shell to core sweetener particles in a granulation or hot melt encapsulation method. Upon cooling and resolidification, the fat tends to have far fewer lesions or micropores, reducing the pathways for moisture to penetrate the treated fat and degrade the sweetener core material. Figure 7 illustrates the procedure used to pressure treat the molten fat or wax and then apply the treated shell material to a sweetener core, such as Aspartame particles.

Figure 2 is a thermograph of a prior art product. It shows that the product (tested using a Dupont Model TA-2000 Differential Scanning Calorimeter) has a mixed melting point profile. This is caused by the instability in the fat material used as the coating on Aspartame. Figure 3 illustrates the thermal profile of the coated Aspartame product of the present invention. Note the single higher melting point, which is characteristic of the Beta Crystalline form in the resolidified pressure treated fat. The material in Figure 3 has greater thermal stability than that of ordinary melted and granulated fat coatings.

An example of the use of fats treated to solidify in the Beta crystalline form, in a hot melt coating method of forming capsules using a sweetener particle as a core is given in Example 4.

Sweeteners may also be encapsulated using traditional encapsulation methods employing natural polymer shell materials, listed in Table 4. Figure 8 illustrates a conventional encapsulation process known as coacervation which may employ such natural polymers.

Figure 1 shows several capsule constructions which may be employed in practicing the present invention which include Reservoir, Micro-Sponge, Multi-Shell and Capsules within Capsules. Reservoir capsule **2** has a shell **4** and a case **6**. Micro-sponge capsule **8** has porus openings **10** in shell layers **12** which encompass core droplets on particles **14**. Encapsulated capsule **16** includes a Reservoir capsule **2** surrounded by a second case **18** which is in turn encompassed by an outer shell **20**. A multi-wall capsule **22** includes a Reservoir capsule **2** with additional shell layers **24**.

Reference is made to the illustration provided in Figure 8, which shows a method of encapsulation known as coacervation, which was specifically designed for use with colloidal or natural polymers. In step one, a core material **26** and shell material **28** are mixed with a volatile organic solvent **30** to form a dispersion **32**. The second step of the process is to add the dispersion **32** to water with a dispersing agent **34** while stirring the agent. The final step is to permit the solvent to evaporate as indicated by numeral **36** while the dispersion is being stirred at a fast speed **35** or a slow speed **37**. Microcapsules **38** or **40** made of core material **26** and shell material **28** are formed. Large capsules **38** or small capsules **40** are formed when stirring occurs at the slow speed **37**, conversely stirring at a fast speed **35** produces smaller capsules **40**.

The following examples illustrate the production of sweeteners using Aspartame as the target sweetener and employing natural polymers processed in a solvent, natural polymers processed in an aqueous media, a combination of two natural polymer shells and a tempered fat shell.

EXAMPLE #1

USE OF A CELLULOSIC MATERIAL AS A SHELL MATERIAL FOR ENCAPSULATED ASPARTAME

This example demonstrates a process for making encapsulated Aspartame using a cellulosic material as a shell coating. The advantage of using a cellulosic material, for Aspartame capsules is apparent when considering the application of those capsules to baked goods, wherein the sugar would be replaced by a bulking agent which then would be used to maintain the integrity of the cake, cookie, pie or other baked good products.

The absence of sugar in cake deflates the size of the cake product. On average, approximately 100-500g of sugar is used to bake a cake. Since Aspartame is approximately 200 times sweeter than sugar, only 1 to 5 grams of Aspartame are be used in the cake formula. A large amount of the volume provided by the sugar must be replaced through the use of a bulking agent. Most bulking agents used commercially in cake, cookies and pies employ the use of a cellulosic compound.

Most such cellulosic compounds are a version of Ethylcellulose or Methylcelloluse. Therefore, to provide compatibility between the Aspartame capsules and the bulking agent used to replace the sugar in cake it was decided to use Ethylcellulose as a coating material for Aspartame. This would then provide compatibility between the Ethylcellulose used as a bulking agent in particular cake formulations and the Ethylcellulose used in the Aspartame capsule.

The Aspartame capsules were produced, according to the following procedure: 20g of Ethocell ST 100 supplied by Dow Chemical Company, was dissolved in 600ml heated Cyclohexane, which was supplied by Aldrich Chemical Company. The Cyclohexane, had been previously heated to 70°C under mild agitation. The mixture of Ethocell and Cyclohexane was stirred until the mixture became clear. Within the Cyclohexane, the Ethocell has formed a film at this point. The mixture was cooled slightly to 60°C and 60g of Aspartame, which was supplied by Tosoh Canada Ltd., was added and the entire mixture was stirred for approximately 1 minute to allow for homogenization.

The apparatus known as the M-CAP #L50 device, using a size control Chamber #2 was employed to actually form the capsules. This apparatus and method of forming capsules is described in United States Patent number 4,978,483. The M-CAP apparatus applies an abrupt pressure change to a mixture of core material and shell material within a liquid dispersion, to form capsules. The device was set for 75 PSI inlet pressure and the mixture of Ethylcellulose, Aspartame and Cyclohexane was processed through the device at a temperature of 60°C for one pass through the device. Capsules were observed to be formed immediately upon exiting the machine and were then later vacuum filtered from the Cyclohexane solvent and dried overnight in a fume exhaust hood. The resulting product consisted of 73.4g of encapsulated product, with a yield of 92%, containing mostly spherical capsules with an average particle size as indicated in Table #6. This sample was known as AES-B-40.

TABLE #6

MATERIAL	AVG. SIZE (M)
Raw APM	29
Gran. APM	650
AES B36	18.18
AES B39	28.33
AES B40	49.33
AES B43.1	107.22
AES A46	4.25
AES A47	4.19
AES A48	4.09
AES A85	3.7

Ethocell coated Aspartame capsules were then applied to cake and cookie formulations, according to cake recipes #1 and #2 and cookie recipes #3 and #4. The results of this baking test indicated that the capsules were well formed and provided for a good dispersion within the cake mix with no void spots, locations which were not sweet, and the cake had no speckles. See Tables #1 and #3. Table #5 indicated the survivability of these capsules after the baking trial. Table #6 shows the average size range of capsules produced according to this method.

08/28/89

Recipe #1

CHOCOLATE CAKE RECIPE
CHOCOLATE CAKE USING ASPARTAME

Formulation: BBF-4

INGREDIENT LIST

Flour	154 gm.
Baking Powder	1.2 gm.
Baking Soda	3.1 gm.
Salt	1.75 gm.
Cocoa Powder	56 gm.
Encapsulated Sweetener	Equivalent to 1.25 gm. of active ingredient
Bulking Agent	198 gm.
Vegetable Oil	56 gm.
Water	92 gm.
Milk	99 gm.
Egg	65 gm.
Vanilla	1.8 gm.

PROCEDURE:

Preheat oven to 350°F, grease and flour one 9x1" x 1/2" round pan. Sift encapsulated sweetener and bulking agent together. Then sift all remaining dry ingredients thoroughly. Place sifted dry ingredients in a mixing bowl and add egg, milk, oil and vanilla. Using a Deluxe Mixmaster Blender, supplied by Sunbeam, blend all ingredients together using medium speed, setting #2, for two minutes. Remove mixing bowl from mixer and scrape excess batter off mixing blades and add to mixing bowl. Stir in ambient water and blend until smooth (batter will be thin) pour into prepared pan. Bake for 30-35 minutes in a 350°F oven or until wood pick inserted in center comes out clean. Remove cake from oven and cool in pan for 10 minutes. Remove from pan and let cool completely.

09/13/89

Recipe #2

YELLOW CAKE RECIPE

YELLOW CAKE USING ASPARTAME

Formulation: J-30 BBF 180-A

INGREDIENT LIST

Encapsulated Sweetener	Equivalent to 1.25 gm. of active ingredient
Bulking Agent	110.2 gm.
Flour	154 gm.
Vanilla	4.2 gm.
Egg	54 gm.
Milk	194 gm.
Shortening	50 gm.
Salt	3.5 gm.
Baking Powder	6.8 gm.

PROCEDURE:

Preheat oven to 350°F, grease and flour one 9x1" x 1/2" round pan. Sift encapsulated sweetener and bulking agent together. Then sift all remaining dry ingredients thoroughly. In a separate bowl, blend milk, egg, shortening and vanilla. Slowly add dry ingredient to the liquid. Using a Deluxe Mixmaster blender, by Sunbeam, blend all ingredients together using medium speed, setting #2 for 3 minutes, scraping side of bowl to insure complete mixing. Remove mixing bowl from mixer and scrape excess batter off mixing blades and add to mixing bowl. Pour into prepared pan. Bake for 20-30 minutes in a 350°F oven or until wood tooth pick inserted in center comes out clean. Remove cake from over and cool in pan for 10 minutes. Remove from pan and let cool completely.

09/23/89

Recipe #3

BUTTER COOKIE RECIPE

BUTTER COOKIE USING ASPARTAME

Formulation: BBF 23A

INGREDIENT LIST

Flour	181 gm.
Encapsulated Sweetener	Equivalent to 1.25 gm. of active ingredient
Butter	85 gm.
Bulking Agent	48 gm.
Vanilla	1.8 gm.
Egg	85 gm.

PROCEDURE:

Preheat oven to 375 deg. F. Sift encapsulated sweetener and bulking agent together. Hand blend sweetening mixture with butter until smooth and creamed (approximately 2 minutes). Add vanilla and egg until well blended. Slowly add in flour until well blended, making sure all flour is mixed homogeneously throughout mixture. Place small chunks of mixture on an ungreased sheet pan and bake for 10 minutes or until cookies are brown around the edges. Remove cookies from sheet pan and let cool.

09/07/89

Recipe #4

OATMEAL COOKIE RECIPE

OATMEAL COOKIE USING ASPARTAME

Formulation: BBF-11-A

INGREDIENT LIST

Shortening	55 gm.
Encapsulated Sweetener	Equivalent to 1.25 gm. of active ingredient
Bulking Agent	110 gm.
Egg	57 gm.
Vanilla	1.8 gm.
Flour	180 gm.
Baking Powder	1.0 gm.
Baking Soda	2.6 gm.
Baking Salt	1.75 gm.
Cinnamon	1.4 gm.
Oats	1 measured cup

PROCEDURE:

Preheat oven at 400°F. In a bowl, mix bulking agent and encapsulated sweetener together, add shortening, egg, vanilla to mixture and blend thoroughly. Slowly mix flour and other dry ingredients together and make sure all dry ingredients are moistened, add oats and blend together for 2 minutes. Place small chunks on ungreased pan and bake 8-10 minutes. Remove cookies from pan and let cool.

TABLE #5

RETAINED ASPARTAME AFTER BAKING IN CHOCOLATE CAKE

	AMOUNT SURVIVED BAKING
RAW ASPARTAME	10%
GRANULATED ASPARTAME	84%
AES-B-36	40.25%
AES-B-39	40.88%
AES-B-40	47.85
AES-B-43.1	65.84

Test performed on aged chocolate cake sample using HPLC apparatus.

In conclusion, Example #1, using a cellulose material as a shell, provided an encapsulated Aspartame which survived the baking process of cakes and cookies, with adequate sweetness throughout, even though only a small quantity of the encapsulated product was used.

EXAMPLE 2

MAKING MICROENCAPSULATED ASPARTAME AND USING A MODIFIED STARCH SHELL PROCESSED USING THE M-CAP DEVICE IN AN AQUEOUS MEDIA

A modified waxy maize starch known as Capsul, supplied by National Starch Company was used as the shell of a sweetener capsule and Aspartame was used as the core sweetener. In this experiment the capsule mix is made in an aqueous media according to the following procedure:

100 grams of Capsul starch material is placed in a glass beaker containing 4 liters of distilled water which has been preheated to 75°C with rapid stirring, until the starch dissolved into a clear solution. The solution was allowed to cool to approximately 50°C until a slight haze formed. 260g

of Aspartame supplied by Tosoh Canada Ltd. was added to the solution over a 15 to 20 minute period of time while still agitating the mixture.

The combined starch-water Aspartame mixture was then passed through the M-CAP device which is the #L50 model, using chamber #2, at 75 PSI, 2 strokes/sec., producing small capsules the size of which is indicated as sample number AES-B-39 in Table #6.

After encapsulation by the M-CAP apparatus the capsules slurry was stirred until the sample reached ambient temperature and was then spray dried Bowen 7 foot spray dryer. The inlet temperature was 290°C, the outlet temperature was 90°C at 8 PSI and 160 ml per hour rate. The fuel rate to the spray dryer was set at a slow rate to avoid burning the starch shell.

The resulting sweetener capsules were mostly spherical with a yield of 60.8%.

The capsules were applied to cake and cookie recipes number 1 through 4 inclusive, and the cake was examined for speckles, sweetness or void spots, and survivability of the Aspartame component after baking. The results are evident in Tables #1, #3 and #5 respectively.

In each case the above made capsules proved superior in resistance to moisture, temperature and Ph degradation during baking, and when applied to cakes and cookies produced a superior baked product.

EXAMPLE 3

EXAMPLE OF SWEETENER THAT HAS BEEN MICROENCAPSULATED USING A MATRIX SHELL COMPOSITION COMPOSED OF TWO NATURAL POLYMERS

Two cellulosic natural materials, Gum Arabic supplied by Tic Gums Company, with a synthetically worked Ethylcellulose, supplied by Berol Chemical, are combined to make a sweetener capsule composed of both natural polymer shells in a single wall matrix.

30g of Gum Arabic and 30g of Ethylcellulose 411, also known as

Bermocell E411 FQ, were blended together and then added to 3,640 ml of distilled water at an ambient temperature in a 4 liter glass beaker under mild agitation.

The mixture was then heated to a temperature of 50°C while stirring over a period of 30 minutes until the solution became clear.

At this point the mixture was cooled to 38°C while still under mild agitation. 260 grams of Aspartame supplied by Tosoh Canada Ltd., was slowly added by spatula. The Aspartame, Gum Arabic, Ethylcellulose and water mixture was stirred 15 minutes before being delivered through the M-CAP Device, Model #L50, with Chamber #2, using 75 PSI inlet pressure, at approximately 2 strokes/sec.

The resultant sweetener capsules were well formed with an average size as indicated in material sample AES-B-36 in Table #6.

The Sweetener capsule mixture, was then spray dried using a Bowen 7 foot spray drier, to obtain free flowing discrete sweetener capsule solids. The total yield was 172 grams or 53.75%.

These capsules were applied to cake and cookie recipes number 1 through 4 inclusive, and the cake was examined for speckles, sweetness or void spots, and survivability of the Aspartame component after baking. The results are evident in Tables #1, #3 and #5 respectively.

In each case, the above made sweetener capsules proved superior in resistance to moisture, temperature and Ph degradation during baking, and when applied to cakes and cookies produced a superior baked product. The use of multiple materials in a single matrix shell composition provides strength to the capsule and greater protection than is possible in some cases where a thin single wall material is employed.

EXAMPLE 4

MAKING SWEETENER CAPSULES CONTAINING A FAT OR WAX SHELL WHEREBY THE FAT OR WAX HAS BEEN TEMPERED USING A THERMAL OR PRESSURE PROCESSING TECHNIQUE

This example illustrates a hot melt process for granulating or coating solid particles using melted fats and waxes. Such processes in the prior art product coatings which are defective and an inadequate barrier against moisture penetration. As shown in Figure 6, this is caused by the formation of cracks or crevices **42** or micropores **44** in the structure of the resultant shell **4** of capsule **2**.

To reduce the formation of cracks, crevices and micropores in fat and wax coatings, the fat must first be tempered into a more stable polymorphic crystalline form. This can be accomplished by thermal or pressure tempering treatments. The result of such tempering treatments is indicated in Figure 6B, where a capsule **2** is shown with a shell **4** with fewer or no cracks or crevices or micropores.

A glycerol tristearin fat in wax form known as Dynasan 118, supplied by Huls of America, with a melting point of 70°C was chosen because of its significantly high melting temperature, offering the potential to be a superior thermal barrier for the targeted sweetener during the baking process. Additionally, it was chosen because of its high purity and crystalline structure.

226 grams of Dynasan 118 was melted in a stainless steel beaker that had been heated to 90°C. The molten fat was then delivered to a pressure applicator device known as the Beta Processor, supplied by Encapsulation Systems, Inc. The Beta Processor applies an abrupt pressure change to a molten fat or wax for the purpose of tempering the fat or wax upon cooling into a resolidified form.

The Beta Processor unit was set at 75 PSI inlet pressure at 1 stroke/sec while the molten Dynasan 118 was passing through the apparatus. The pressure treated fat/wax was collected and was then applied while still in a molten state to 75 grams of Aspartame, supplied by Tosoh Canada Ltd., in a mixing bowl of an orbital mixer supplied by Hobart Company.

The mix was agitated until the temperature cooled in the Dynasan 118/Aspartame mixture to 55°C, at which point the fat shell resolidified into a complete coating over the Aspartame particles, thereby forming sweetener capsules.

The resulting white solid product had a yield of 98%. There were no solvents used in this process. This sample is identified as AES-B-43.1 in Table #6, the size scan comparison.

These capsules were applied to cake and cookie recipes number 1 through 4 inclusive, and the cake was examined for speckles, sweetness or void spots, and survivability of the Aspartame component after baking. The results are evident in Tables #1, #3 and #5 respectively.

In each case, the sweetener capsules made in the above Example 4 proved superior in resistance to moisture, temperature and Ph degradation during baking, and when applied to cakes and cookies produced superior baked goods.

The DSC spectra evident in Figure 3 is superior to the DSC spectra illustrated in Figure 2 which is Aspartame coated with a simple, untempered fat formulation obtained from Nutrasweet Company. Note the higher melting point and more stable crystalline structure of the Beta crystalline form fat/wax of Figure 3 compared to the instability and lower melting point of the fat DSC spectra of Figure 2. The untempered fat illustrated in Figure 2 has a less defined melting point and, under microscopic examination, is found to contain several micropores, cracks and crevices, as shown in Figure 6A, thereby having reduced resistance to moisture penetration. Since Aspartame is Ph sensitive as well as heat sensitive, moisture penetration through cracks in the fat shell coating causes rapid decomposition of the Aspartame's active ingredient producing decomposition products which are illustrated on Figure 4, and reducing the sweetness potency. The decomposition products include Phenylalanine, Diketopiperazine Aspartic Acid and Aspartylphenylalanine.

The pressure treated fat which results in the Beta crystalline form have far fewer cracks or micropores, as may be observed through the illustration provided in Figure 6B, thereby resisting moisture penetration and the Ph difficulties accompanying moisture penetration. Additionally, tempered fat shell has a higher more stable melting point enabling resistance to heat during the baking stage, as seen in Figure 3. Because a fat is used as the shell, the fat tends to immediately release the sweetener upon contact in the mouth. This enables the sweetness to be sensed instantly upon the first bite of the baked goods incorporating the sweetener capsules.

The samples made from the above experiments were tested for survivability in chocolate cake made according to Recipe 1, using a HPLC test procedure which compared the amount of Aspartame capsules fed into the cake, equal to 1.25 grams of active material in each test Recipe, to the amount of Aspartame found in the cake sample after baking, and after the cake has been aged for at least 5 days. The results of this study are listed in Table 5, where it can be seen that the granulated, large particle sized Aspartame product has the highest retention and, therefore, the least generation of decomposition by-products. However, this product also leads to a speckled appearance in cake and to several void spots because of its size and accompanying poor distribution throughout the dough. The other encapsulated samples made according to the procedures listed above all have acceptable retention of the Aspartame.

It should also be noted that when using an encapsulated sweetener in a baked good, the customer expects instant gratification upon the first taste of the baked product. If the capsule protects the sweetener from the degradating effects of dough preparation or baking, but does not allow instant sensing of sweetness upon the first taste by a customer, the encapsulation effort is wasted. Most Reservoir type capsules, as shown in Figure 1, would never release the sweetener if they were designed to be totally secure enclosures, until the capsules were actually digested. To provide an instant gratification to the customer, the capsules should ideally provide protection of the sweetener core throughout the dough preparation and baking stages, but allow release of the sweetener at the very end of the baking cycle. In this manner, the capsule has released, or has had at least a partial dissolution of the capsule shell leaving the sweetener core exposed, at the end of the baking cycle, so that there is an immediate sweet taste sensation upon first bite. The particular coating materials listed in Table #4 were selected because they would offer protection during the baking stage but would partially release toward the end of the baking cycle, enabling instant sweet gratification upon first bite.

Although sweetness is a subjective thing, open to different perceptions by various people, the scale developed in Table #3A, shows the perceptions of a test audience composed of 20 persons when taste testing various cake and cookie formulations. A comparison was made of cakes made with no Aspartame, raw Aspartame, Granulated Aspartame and each of the examples of encapsulated Aspartame listed herein. Note that in each case the encapsulated Aspartame was superior. Table #3B indicates that during the taste test the baked goods tested had little or not aftertaste when the encapsulated Aspartame was used, and a severe aftertaste when the raw or the granulated Aspartame was used.

Various bulking agents may be used to replace the bulk when using high potent sweeteners in place of the normal sugar bulk. In the particular case of Aspartame, which is nearly 200 times sweeter than sugar, only 1 to 2 grams of Aspartame active material may be applied to a conventional cake formation, which would normally contain as much as 100 to 200 grams of sugar. The lost bulk, approximately 198 grams by weight, must be replaced by using a bulking agent. Most bulking agents are cellulosic compounds which, when used in large quantities as is required in the use of Aspartame, can offer laxative problems. Certain bulking agents do not offer laxative problems; one such bulking agent is a product known as Snowite Oat Fiber supplied by Canadian Harvest Company. Additionally, it is important to consider some advantages in using bulking agents. Bulking agents control body or viscosity, and provide smooth, stable textures which improve mouth feel. Most bulking agents are soluble in cold water, improve browning and provide low to moderate sweetness with no starch flavors. One of the biggest advantages in using bulking agents is caloric reduction; however, few bulking agents claim to reduce calories.

The Aspartame capsules of the present invention has shells of a fat or wax having at least two crystalline forms. The capsules shells are formed so that they are in the Beta crystalline form, which forms a more homogenous shell which is more resistant to moisture, temperature and Ph degradation during baking. The present invention treats the fat or wax by an abrupt pressure change to the molten fat or wax; this causes it to solidify into the Beta crystalline form as opposed to its normal Alpha crystalline form.

The shell material can be treated and then used to form capsules using a conventional method, as in Example 4 where a hot melt process was used. In Examples 1-3 the shell material is treated as the capsules are formed using the M-CAP method referred to above.

Statement of Industrial Utility

The encapsulated sweeteners of the present invention offer improved survival during baking, thus permitting the use of non-sugar sweeteners in baked goods, such as cakes and cookies.

WE CLAIM:

1. A method of making sweetener capsules comprising the following steps:

(a) forming a dispersion of a core material composed of a sweetener and a shall material; and

(b) applying an abrupt pressure change to the dispersion.

2. A method of making capsules according to Claim 1 wherein the sweetener core material is composed of natural sweeteners, artificial sweeteners or a combination of both natural and artificial sweeteners.

3. A method of making capsules according to Claim 1 wherein the sweetener core material is selected from the group of Aspartame, Saccharin, Nutrasweet, ACE-K (Sunett), Cyclamate, Surolose, Alitame, Left Handed Sugars in either liquid or solid form.

4. A method of making capsules according to Claim 1 wherein the sweetener core material is selected from the group of Sucrose, Glucose, Lactose, Corn Syrup, Corn Syrup Solids, Dextrose, Fructose and Mannitol.

5. A method of making capsules according to Claim 1, wherein the sweetener core material comprises a blend of both natural and artificial sweetener compounds.

6. A method of making capsules with a fat or wax shell, comprising the following steps:

(a) melting the fat or wax into a molten state;

(b) treating the molten fat or wax with either heat or the use of abrupt pressure changes to temper the fat or wax;

(c) applying the treated molten fat or wax to sweetener particles in a vessel and then agitating the mixture until such time as the treated fat or wax solidifies into a coating over the sweetener particle; and,

(d) removing the treated or tempered fat or wax coated sweetener from said vessel.

7. A means of making sweetener capsules according to Claim 6

wherein the fats or waxes which are employed as tempered coatings are selected from the group of Cotton Flakes, Glycerol, Tristearin, Carnoloa Wax, fats or waxes derived from vegetable products, insects and animal or synthetic products.

8. A means of making sweetener capsules according to Claim 1, wherein the resultant size of the capsules is under 150 microns.

9. A means of making sweetener capsules according to Claim 6, wherein the resultant size of the capsules is under 150 microns.

10. A food product, such as cookies, pies, snack food, rolls, cakes, muffins, bread, candy or any other food which is heated, baked, fried or microwaved, wherein a bulking agent, composed of dietary fiber is employed as a substitute for sugar, and is used in conjunction with encapsulated sweeteners made by the method described in Claim 1.

11. A food product, such as cookies, pies, snack food, rolls, cakes, muffins, bread, candy or any other food which is heated, baked, fried or microwaved, wherein a bulking agent, composed of dietary fiber, is employed as a substitute for sugar, and is used in conjunction with sweetener capsules made by the method described in Claim 5.

12. Means of making sweetener capsules according to Claim 1, wherein multiple shell materials are employed.

13. Means of making capsules according to Claim 1, wherein a blend of multiple shell materials is employed.

14. Means of making capsules, according to Claim 6, wherein a blend of different fats or waxes is employed as a combination shell material.

15. A means of providing thermal stability for sweetener capsules according to Claim 6, wherein tempering is used to provide a higher melting point in the treated fat or wax coating.

16. A means of providing Ph and moisture resistance for sweetener capsules according to Claim 6, wherein tempering is used to help reduce the number of micropores or crevices on the treated fat or wax coating.

17. A means of reducing the aftertaste of certain sweeteners, particularly Aspartame, through the process of encapsulation, according to Claim 1.

18. A means of making sweetener capsules according to Claim 1, wherein the sweetener capsules are recycled through the encapsulation process more than once.

19. A means of making sweetener capsules according to Claim 18, wherein the recycling of the capsules is used to produce capsules inside of a larger capsule enclosure.

20. A means of making sweetener capsules according to Claim 18, wherein the recycling of the capsules is part of a size reduction process.

21. A means of making sweetener capsules according to Claim 18, wherein the recycling of the capsules is part of an effort to provide additional shell layers, which may or may not be composed of the original starting shell material.

22. Capsules containing sweeteners produced according to the method of Claim 1 which are characterized by:

(a) sizes under 150 microns;

(b) free-flowing capsule powders which provide homogeneous distribution in the starting ingredients of various baked goods;

(c) an ability to protect the sweetener from Ph, moisture and the baking temperatures present in the formation of the final baked goods;

(d) an ability to retard decomposition of the sweetener's active ingredient during the baking process by providing a protective enclosure for the sweetener's active core material;

(e) an ability to survive the majority of time during which the sweetener capsules will be exposed to heat during the baking process, but to eventually effect partial or total dissolution of the shell toward the end of the baking cycle, thereby providing an immediate sweet sensation upon the first bite by a customer into the baked good; and

(f) an ability to reduce the aftertaste, if any, associated with the use of the sweetener in either its raw form or in a form produced during thermal exposure through the protections offered by a capsule shell enclosure.

23. A method for making sweetener capsules using the method of Claim 1, employing the use of cellulosic shell materials selected from the group of nitrocellulose, ethylcellulose, methylcellulose or a combination of these cellulosic compounds.

24. A method for making sweetener capsules according to the method of Claim 1, employing natural polymers selected from the group of Gum Arabic, Proteins, Gelatin, Modified or Unmodified Corn Starch, Cellulose Acetate Phthalate, Arabinogelactan or Propylhydroky Cellulose.

TYPES OF MICROCAPSULES

RESERVOIR CAPSULE: SINGLE FILL

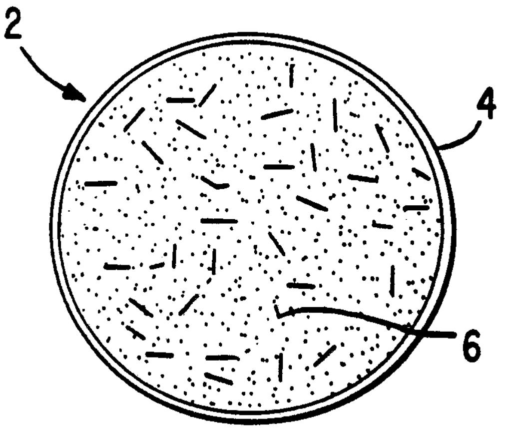


FIG. 1A

MICRO-SPONGE CAPSULE

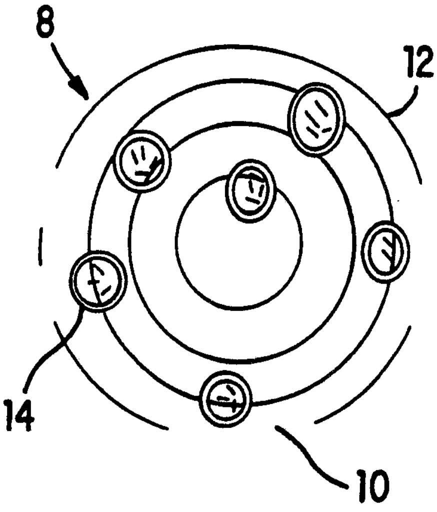


FIG. 1B

MULTI-FILL CAPSULE: CAPSULES WITHIN CAPSULES

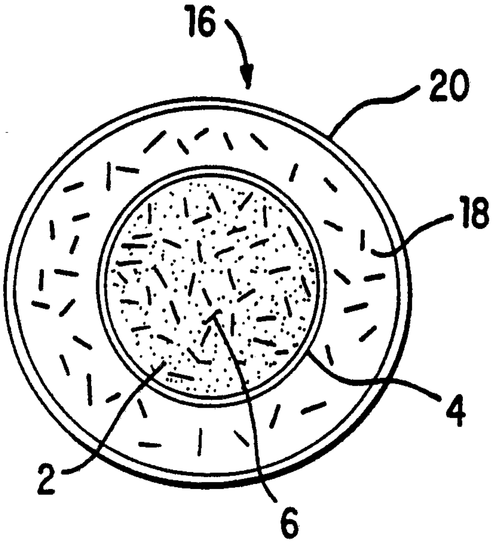


FIG. 1C

MULTI-WALL CAPSULES

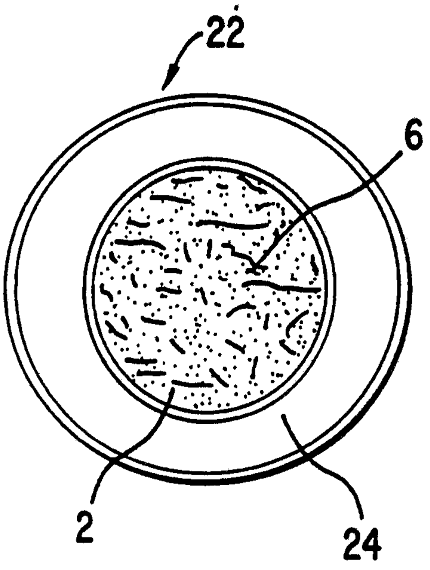


FIG. 1D

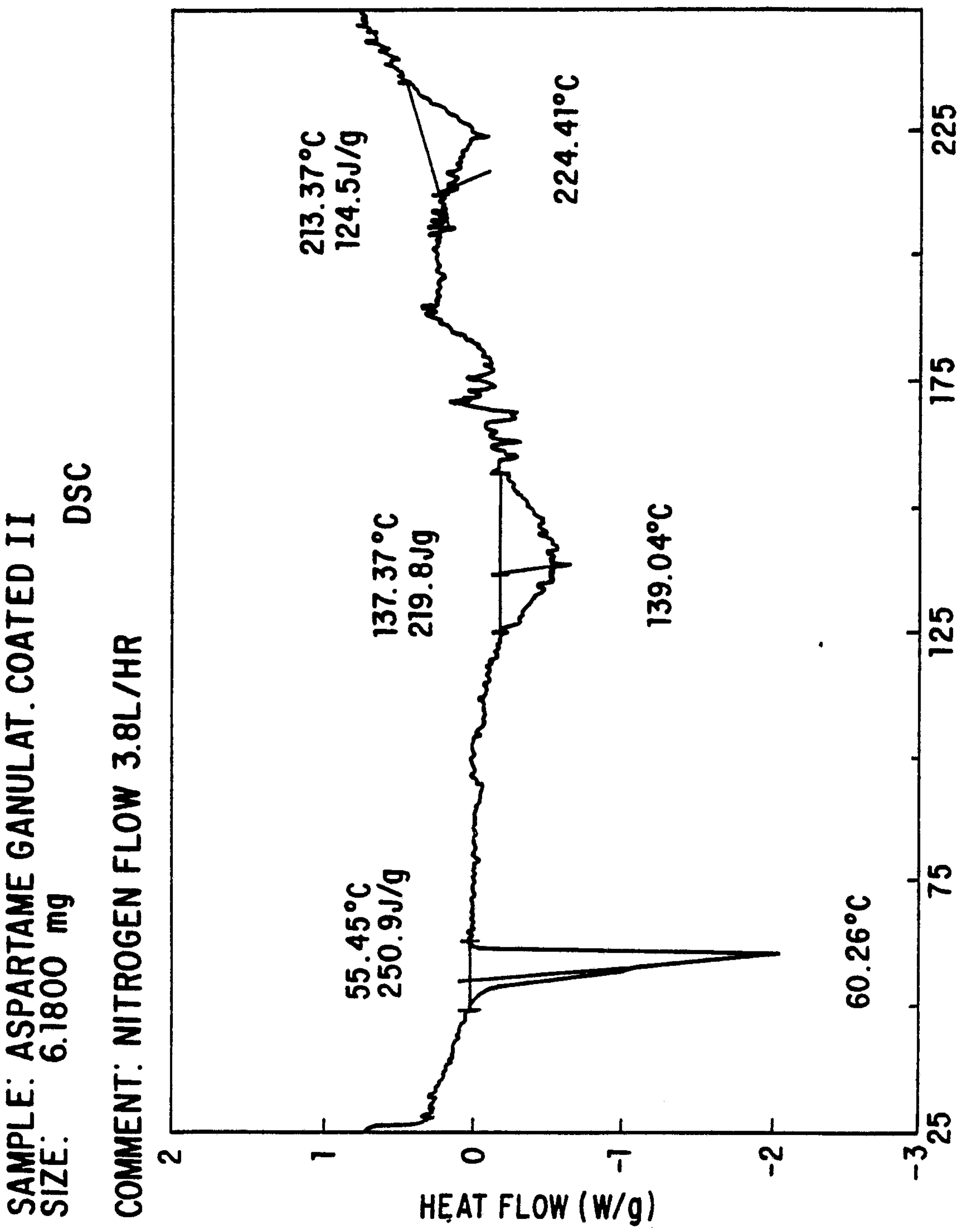

SAMPLE: ASPARTAME GANULAT. COATED II
SIZE: 6.1800 mg
DSC
COMMENT: NITROGEN FLOW 3.8L/HR
55.45°C
250.9J/g
60.26°C
137.37°C
219.8Jg
139.04°C
213.37°C
124.5J/g
224.41°C
2
1
0
−1
−2
−3
HEAT FLOW (W/g)
25
75
125
175
225

FIG. 2

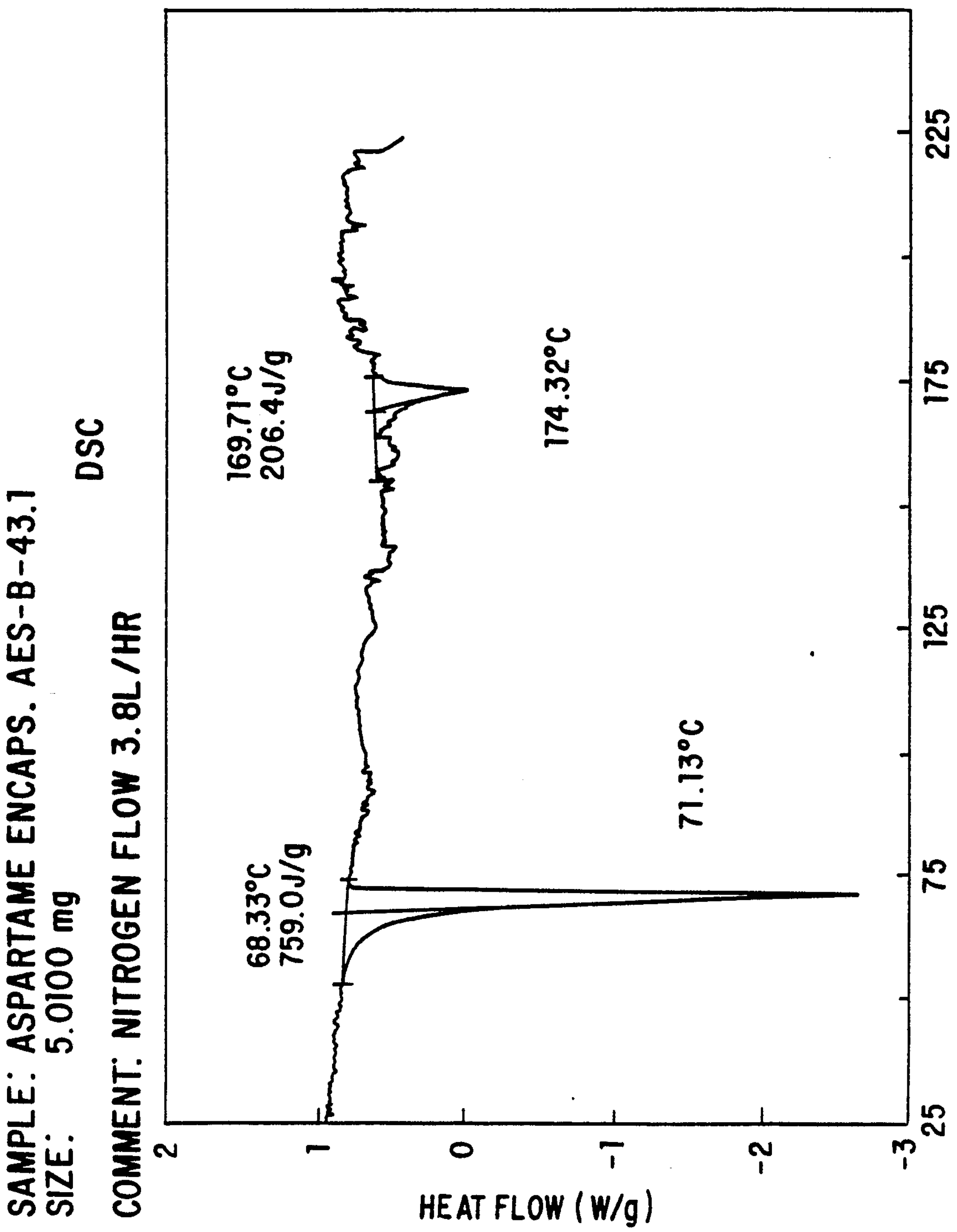

SAMPLE: ASPARTAME ENCAPS. AES-B-43.1
SIZE: 5.0100 mg
COMMENT: NITROGEN FLOW 3.8L/HR
DSC
169.71°C
206.4J/g
174.32°C
68.33°C
759.0J/g
71.13°C
HEAT FLOW (W/g)
2
1
0
-1
-2
-3
25
75
125
175
225

FIG. 3

ASPARTAME DECOMPOSITION PRODUCTS
FROM MOISTURE AND HEAT

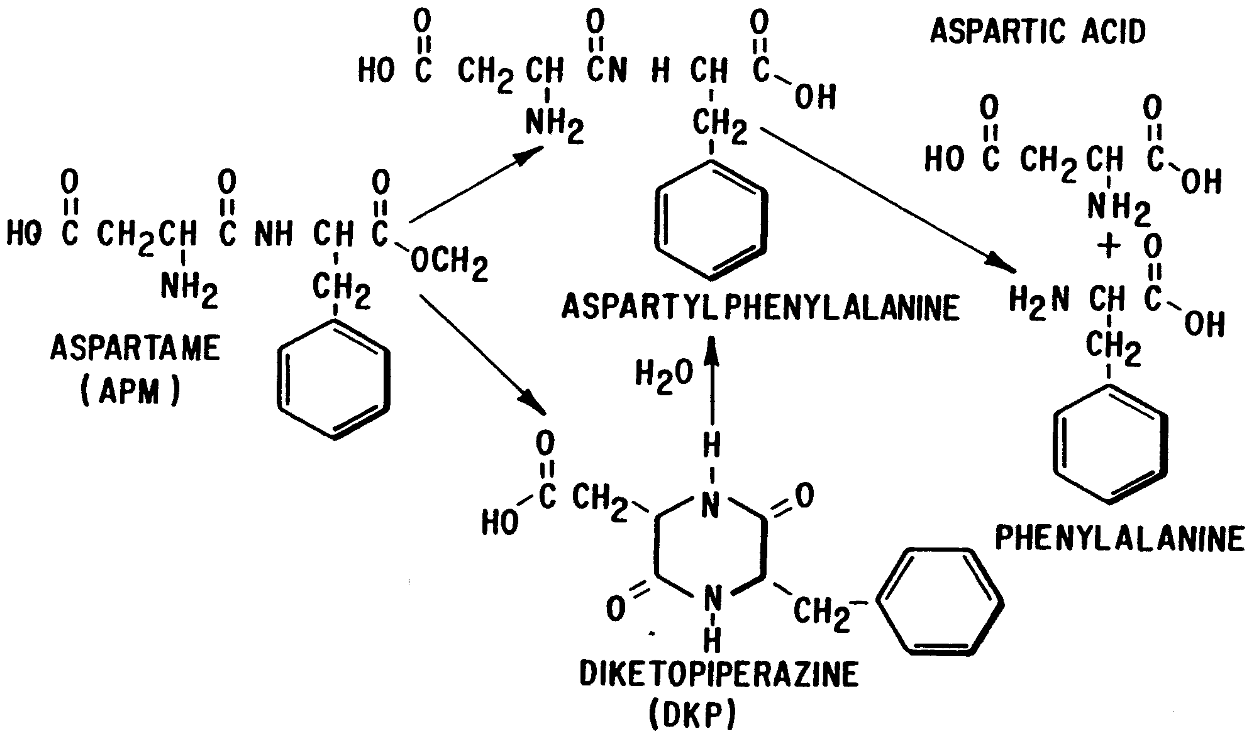


FIG. 4

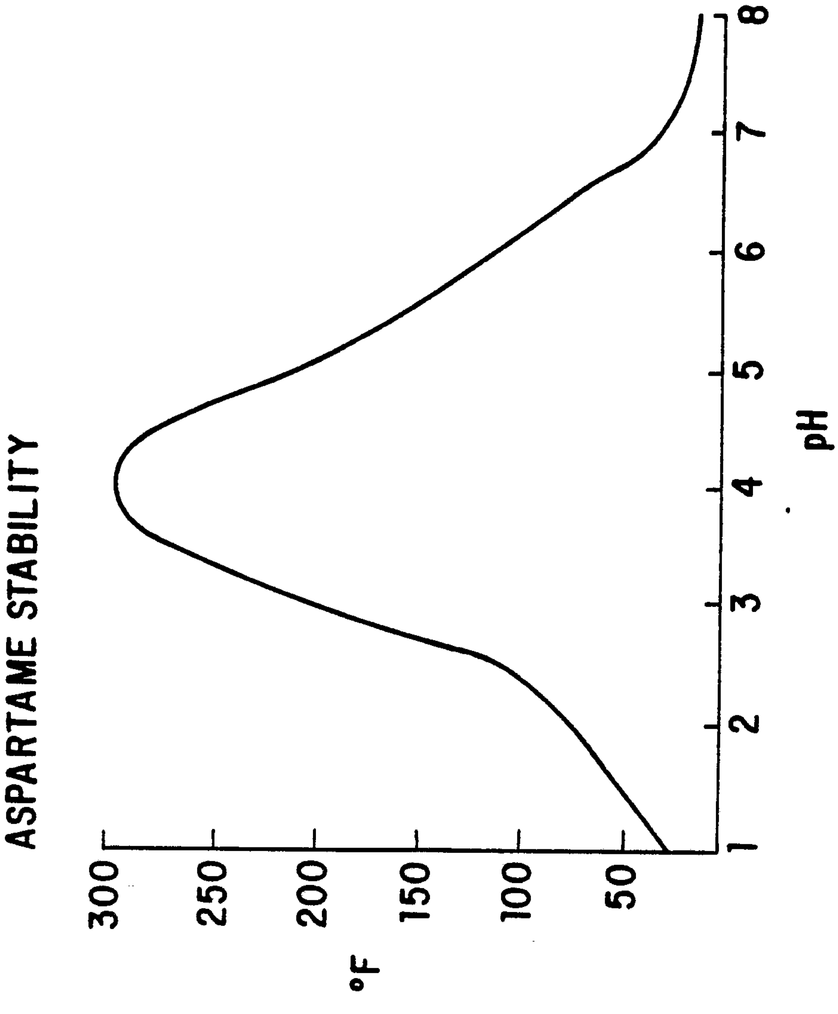


FIG. 5

NORMAL FAT/WAX COATING APPLIED TO A SOLID PARTICULATE BY HOT MELT AND GRANULATION PROCESS

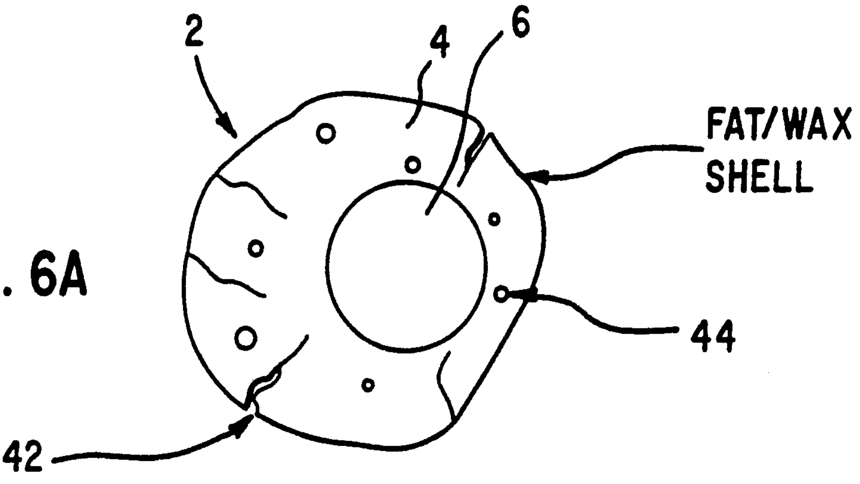


FIG. 6A

BETA STABLE FAT/WAX COATING WITH LITTLE OR NO MICROPORES OR CREVICES

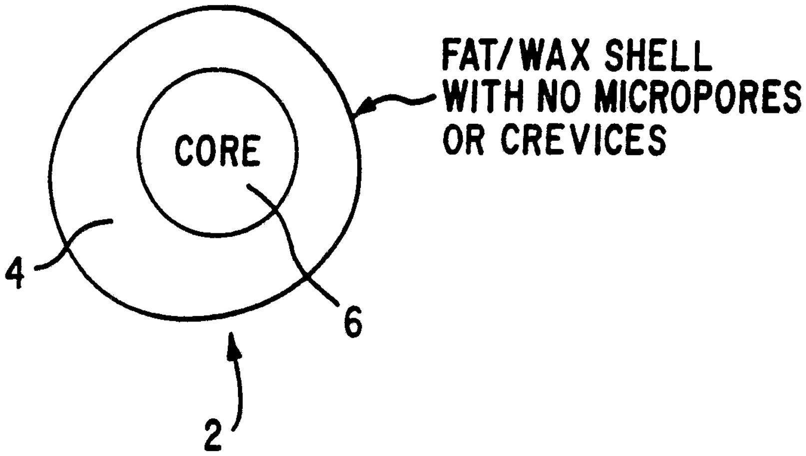
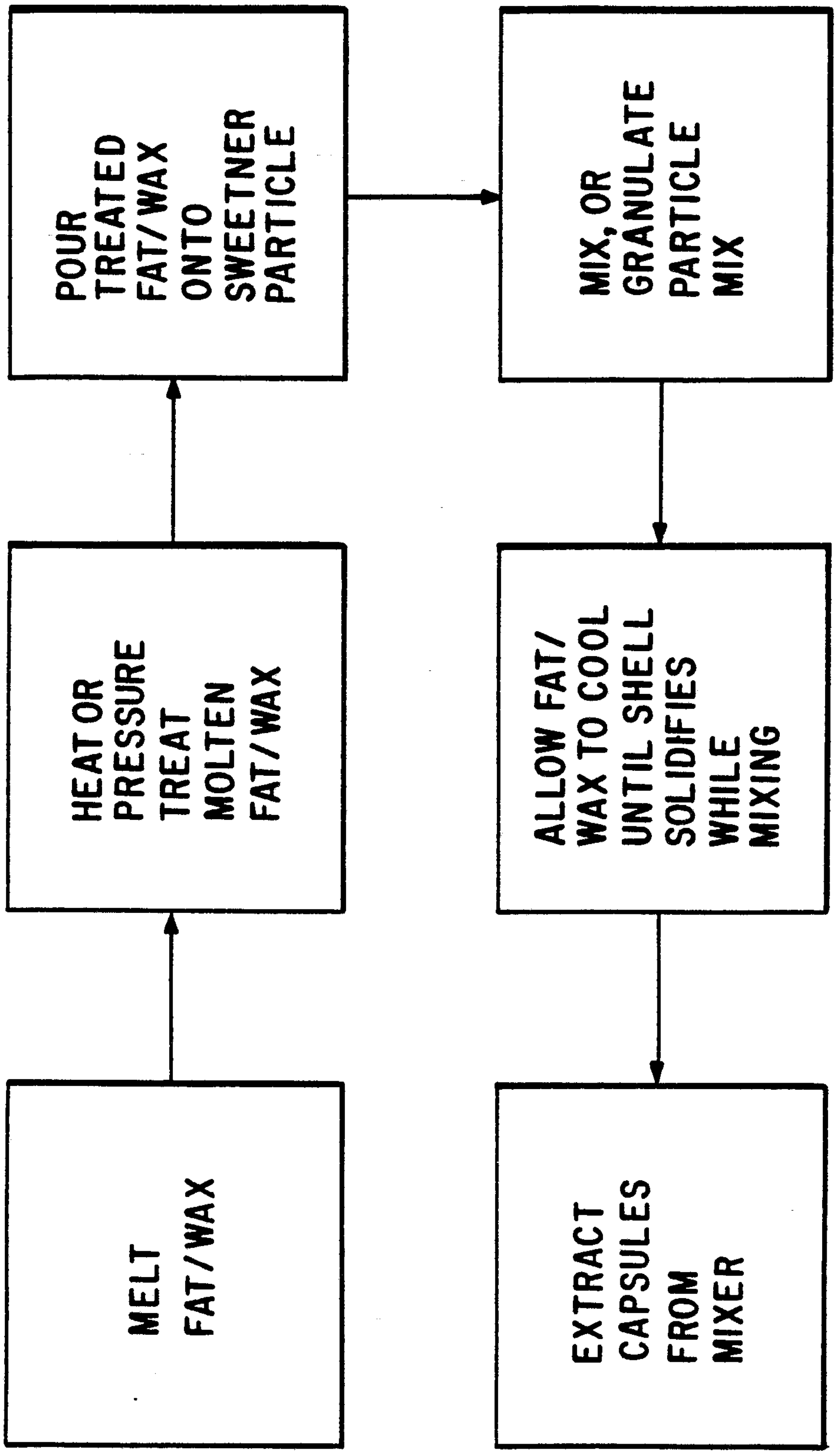


FIG. 6B

FIG. 7

BETA TREATED FAT/WAX



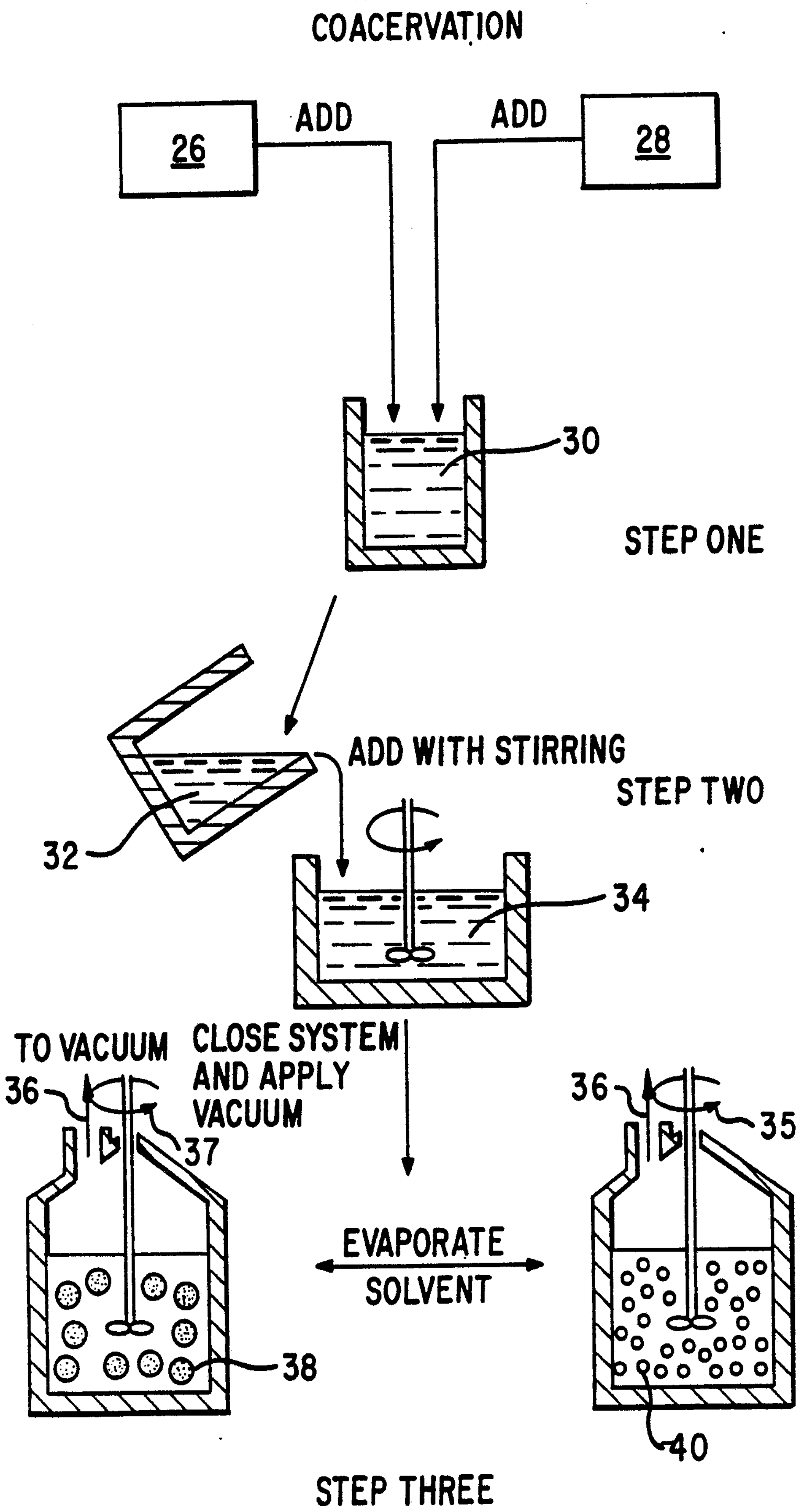


FIG. 8 PRIOR ART

INTERNATIONAL SEARCH REPORT

International Application No. PCT/US91/09434

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [6]

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC(5): B01J 13/04, 13/20, 13/22; A23L 1/216, A23L 1/236

II. FIELDS SEARCHED

Minimum Documentation Searched [7]

Classification System	Classification Symbols
U. S.	264/4.1; 427/213.3, 427/213.31; 428/402.24; 426/96, 426/548

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [8]

APS Text Search

III. DOCUMENTS CONSIDERED TO BE RELEVANT [9]

Category *	Citation of Document, [11] with indication, where appropriate, of the relevant passages [12]	Relevant to Claim No. [13]
&, P	US, A, 4,978,483 (REDDING, JR.) 18 DECEMBER 1990.	1-9, 12-21, 23, 24
A	US, A, 4,704,288 (TSAU ET. AL.) 03 NOVEMBER 1987	6,7,9,14-16
A	US, A, 4,384,004 (CEA ET. AL.) 17 MAY 1983.	
A	US, A, 3,962,468 (PISCHKE ET. AL.) 08 JUNE 1976.	
A	US, A, 4,122,195 (BAHOSHY ET. AL.) 24 OCTOBER 1978 See Column 4.	
A	US, A, 4,230,687 (SAIR ET. AL.) 28 OCTOBER 1980.	1
A	US, A, 4,465,694 (OKADA) 14 AUGUST 1984. See Columns 3 and 4.	
A	US, A, 4,515,769 (MERRITT ET. AL.) 07 MAY 1985.	
A	US, A, 4,740,376 (YANG) 26 APRIL 1988.	
A	US, A, 4,804,548 (SHARMA ET AL.) 14 FEBRUARY 1989	6,7,9,14-16
X Y	US, A, 4,824,681 (SCHOBEL ET. AL.) 25 APRIL 1989	10, 11, 22 10, 11, 22
X Y	US, A, 4,933,190 (CHERUKURI ET. AL.) 12 JUNE 1990. See Column 5 and 6.	10, 11, 22 10, 11, 22
P,Y	US, A, 4,997,659 (YATKA ET. AL.) 05 MARCH 1991. See See Columns 1-4.	10, 11, 22

* Special categories of cited documents: [10]

"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

"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

IV. CERTIFICATION

Date of the Actual Completion of the International Search 17 MARCH 1992	Date of Mailing of this International Search Report 09 APR 1992
International Searching Authority IPEA/US	Signature of Authorized Of NGUYEN NGOC-HO INTERNATIONAL DIVISION For JOHN M. COVERT Ngoc Ho Nguyen