

PATENT SPECIFICATION

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(54) NICOTINE FORTIFICATION OF SMOKING PRODUCTS

(71) We, IMPERIAL GROUP LIMITED, formerly Imperial Tobacco Group Limited, a British Company of Imperial House, 1 Grosvenor Place, London SW1X 7HB, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention concerns a process for making a smoking material e.g. tobacco, hand rolling tobacco, pipe tobacco, reconstituted tobacco sheet, substitute smoking materials free from tobacco, or blends and mixtures thereof and cigarettes, cigars or cigarillos containing such materials and concerns the problem of maintaining or increasing the nicotine content of the smoke whilst avoiding an undesirable level of particulate matter in the smoke from the product.

Various techniques are known for reducing particulate matter in the smoke of a smoking product. For example it is well known to filter out particulate matter but in so doing nicotine is also filtered and thus the resulting smoke from the smoking product is poorer in nicotine as well as in particulate matter.

A further example is the use of an acceptable smoking base material such as reconstituted tobacco sheet in blends for smoking products. By appropriate choice of component materials and control in the preparation and manufacture of the sheet it is possible to reduce the yield of particulate matter in the resulting smoking product. Unfortunately such sheet, prepared from mixtures of tobacco stem, tobacco lamina and/or general tobacco offals, generally contains a lower concentration of nicotine than the other tobacco grades in the blend such that a nicotine deficiency exists.

A third example is the use of acceptable synthetic and substitute smokable base materials in which particulate matter is reduced considerably. As with the previous examples however, the overall delivery of

nicotine in the smoke is reduced in proportion to the amount of such material incorporated into the smoking product since the natural nicotine content of such smoking materials is zero.

To avoid the disadvantages of the above examples it is proposed to increase the nicotine content of a smoking product whereby, although the level of particulate matter can be reduced, the level of nicotine can be maintained or even increased. Efforts to do this in the past have been disappointing since the percentage of nicotine added to the product and subsequently appearing in the smoke has been very low. Furthermore the level of the added nicotine drops considerably with time and shelf life is poor. It is an object of the present invention to overcome such disadvantages.

According to the present invention there is provided a process for making a smoking material comprising adding to a smokable base material a nicotine derivative of a polyuronic acid, whereby on pyrolysis of the resulting smoking material controlled amounts of nicotine are released into the smoke.

Preferably the polyuronic acid is a polygalacturonic or polymannuronic acid and the derivative is nicotine pectinate or nicotine alginate respectively.

Where nicotine pectinate is used this is preferably formed from pectinic acid having a degree of esterification of the order of 30%—35%. Furthermore the acidity is from 1.5 to 3.5 milli equivalents per gram, preferably from 2.0 to 3.5 milli equivalents per gram (m.eq/g).

If pectinic or alginic acids available are not of the desired acidity, they can be dashed or freed from inorganic materials by washing with acid, preferably with aqueous isopropanol (specific gravity 0.880) containing hydrochloric acid in the ratio of 1 part by volume of hydrochloric acid to 20 parts by volume of aqueous isopropanol (specific gravity 0.880).

The acceptable smoking base material can be tobacco lamina, stem (mid rib), reconstituted sheet or synthetic smoking material which is tobacco free.

- 5 Preferably, 3 to 13% of the nicotine derivative on a dry weight basis is added to the base material.

- 10 The invention is illustrated by the following two series of examples. For use in these series the following samples of nicotine alginate and nicotine pectinate were produced:—

- 15 A first sample (Sample 1) of nicotine pectinate was produced from citrus pectinic acid obtained from lemon peel and having a degree of esterification of 35% and acidity of 3.1 m.eq/g and 100 gms of this acid was mixed with 30 gms of water until the water was absorbed and a free-running powder was obtained. 50gms of 95% pure technical grade nicotine was stirred into the powder to produce an homogeneous paste of crude nicotine pectinate. This was left 24 hours to complete the chemical reaction and the resulting product was ground to a coarse powder. One litre of ethanol was added to the powder which was filtered, washed with five 100ml portions of ethanol and two 50 mls portions of diethyl ether and dried so that unreacted nicotine was removed. The resulting powder had a nicotine content of 25%. This nicotine pectinate will hereinafter be referred to as "Sample 1".

- 35 A second sample — hereinafter referred to as "Sample 2" — of nicotine pectinate was prepared from citrus pectinic acid prepared from lemon peel and having a degree of esterification of 35% and acidity of 2.7 m.eq/g. With this pectinic acid, nicotine pectinate was prepared in exactly the same way as that set out above with reference to sample 1 but the resulting nicotine pectinate had a nicotine content of 23.8%.

- 40 A powder of nicotine alginate (Sample 3) was prepared using alginic acid of acidity 5.2 m.eq/g supplied by British Drug Houses Ltd. of Poole, Dorset under Reference 27898 in their 1970 Catalogue. Nicotine alginate was prepared from this alginic acid in exactly the same way as Samples 1 and 2 of nicotine pectinate were prepared and as described above, and the resulting nicotine alginate had a nicotine content of 31.5%.

- 55 A fourth sample — Sample 4 — of nicotine pectinate was prepared in solution form but in this case technical grade nicotine 90.8% pure and supplied by British Drug Houses was used with lemon peel pectinic acid having a degree of esterification of 35% and acidity of 2.7 m.eq/g. A solution of 1200 g of pectinic acid in 18.5 litres of water was prepared and 333g of nicotine added. The dissolved nicotine pectinate derivative contained 20% nicotine and the final solution contained 1.5% nicotine on a weight for weight basis.

- 65 A fifth sample — Sample 5 — of nicotine

pectinate was prepared according to the procedure of sample 4 but in this case a sugar beet pectinic acid was used having a degree of esterification of 30% and an acidity of 1.7 m.eq/g. The resulting nicotine pectinate solution had a nicotine content of 1.5% on a weight for weight basis.

Sample 6 was a nicotine alginate solution made according to the method of sample 4 above but in this case the acidity of the alginic acid was 3.6 m.eq/g with a negligible degree of esterification. The nicotine alginate solution had a nicotine content of 1.5% on a weight for weight basis.

Sample 7 was a nicotine pectinate solution prepared from the same materials as sample 4 except that 15 litres of water was used instead of 18.5 litres. The resulting nicotine pectinate solution contained 1.8% nicotine on a weight for weight basis.

The first series of experiments was conducted using 9 examples, of which examples 1—6 involved fortification using nicotine pectinate or nicotine alginate and examples 7—9 were controls or standards. The second series involved 11 examples of which numbers 10—17 were fortified with nicotine alginate or nicotine pectinate and examples 18—20 were controls or standards.

Series 1: Examples 1—9.

Example 1.

A sheet of reconstituted tobacco was prepared in the following manner.

A mixture of 225gms of tobacco winnowings and 225gms of general tobacco offal (both oven dry weight) were soaked in 4.5 litres of tap water for one hour. The slurry was repeatedly passed through a Premier 84 colloid mill initially set to a gap of 0.01 inches between the stones. The gap was decreased by 0.003 inches after each pass until the slurry remained in a colloidal state.

20gms of Sample 1 nicotine pectinate were added to the tobacco slurry and mixed using a Silverson mixer. The mixture was then passed through the colloid mill set at gap zero to remove any lumps which may have formed due to coagulation.

The slurry so formed was cast onto a pre-siliconed stainless steel sheet to a wet sheet thickness of 1.2mm. The sheet was dried in air and then slightly moistened to facilitate removal from the stainless steel sheet. The tobacco sheet so prepared was tested for nicotine content, cut and plain cigarettes of 2.5mm circumference and 70mm length (commonly referred to in U.K. as "class B" cigarettes) were manufactured on a Hauni Baby cigarette making machine and smoked using a standard smoking machine procedure in which five equal weight cigarettes are smoked on a Filtrona CSM 14 machine to a 20mm butt using a 2 second, 35ml puff every minute. (Filtrona is a Registered Trade Mark). The

particulate matter was collected on a Cambridge glass fibre filter and the water-free and nicotine-free particulate matter (P.M.) was measured and the nicotine delivery determined. To do this, the wet deposit on the Cambridge filter was weighed, the nicotine was determined by steam distillation followed by spectrophotometric analysis (Willits method) and water determined by gas chromatography. The resultant deposit is particulate matter (P.M.) which is water and nicotine free. The tests were repeated three months later and the results are tabulated on the accompanying Table 1.

Example 2.

A sheet of reconstituted tobacco was prepared in exactly the same way as that described above with reference to Example 1 and nicotine pectinate of Sample 1 was again added but in this example 36.3gms of the nicotine pectinate were added to the same tobacco slurry.

The cast sheet was removed from the band, tested for nicotine content cut and made into plain cigarettes 25mm circumference and 70mm long which were subsequently tested by the standard method described in Example 1 for particulate matter and nicotine deliveries in the smoke. The tests were carried out soon after manufacture and three months later and the results are set out in Table 1.

Example 3.

A sheet of reconstituted tobacco was again manufactured in accordance with the details set out in Example 1 above, but in this case Sample 2 of the nicotine pectinate was added to the slurry, 21.4gms of the pectinate being used for the same amount of the tobacco slurry. Again, nicotine content of the sheet was measured, the sheet was cut, made into plain cigarettes of 25mm circumference and 70mm length and analysed by the standard method described in Example 1 for particulate matter and nicotine deliveries in the smoke both immediately after manufacture and three months later. The results are set out in Table 1.

Example 4.

A sheet of reconstituted tobacco was made as described above, with reference to Example 3 but the amount of Sample 2 nicotine pectinate added in this instance was 47.7gms for the same amount of tobacco slurry. Again after determining its nicotine content the sheet was cut and made into plain cigarettes 25mm circumference and 70mm long and subsequently analysed by the standard method described in Example 1 for particulate matter and nicotine deliveries in the smoke. After an interval of three months the smoke analysis was repeated.

Example 5.

The sheet of reconstituted tobacco des-

cribed above with reference to Example 1 was again made strictly in accordance with the specification but 16.2gms of the nicotine alginate (Sample 3) were added to the tobacco slurry. Again the sheet was cut and made into plain cigarettes 25mm circumference and 70mm long. These were tested by the standard method described in Example 1 for particulate matter and nicotine deliveries in the smoke and the results are set out in Table 1.

Example 6.

The sheet of example 5 was made up but 26gms of Sample 3 nicotine alginate were added and the resulting sheet cut, made into plain cigarettes 25mm circumference and 70mm long and tested by the standard method of example 1 for particulate matter and nicotine delivery in the smoke.

Example 7.

To provide one control against which one could analyse the results of storage of the samples set out above, a sample of a commercially available low tar yielding reconstituted tobacco sheet of nicotine content 0.78% was made into plain cigarettes 25mm circumference and 70mm length and tested by the standard method of example 1 for particulate matter and nicotine delivery in the smoke on the same dates as examples 1 to 6 were tested. This particular control sheet is recognised as being relatively stable for nicotine content and thus provided a suitable standard against which to measure the nicotine loss from the samples 1 to 6.

Example 8.

The tobacco sheet described above with reference to examples 1-6 was made but without any additive i.e. no nicotine pectinate or nicotine alginate, simply tobacco winnowings and offal with water. The non-additive sheet so made was cast on the stainless steel band and, when removed, was cut, made into plain cigarettes 25mm circumference and 70mm long and analysed by the standard method of example 1 for particulate matter and nicotine delivery in the smoke.

Example 9.

To provide a comparison and standard by which the result set out in Table 1 can be compared, a plain cigarette 25mm circumference and 70mm long and available on the U.K. market was obtained and analysed in the same way as the other examples for particulate matter and nicotine delivery in the smoke and the results are set out in Table 1.

Table 1.

Referring to Table 1 it will be noted that the amount of nicotine in the smoke in all

5 examples 1—6 was appreciably higher than
10 in any of the three control samples 7, 8 and
9. The percentage nicotine transfer (nicotine
in smoke compared with nicotine in unsmoked
cigarette) was better in examples 1—6 com-
pared with examples 7 and 9. Furthermore
the ratio of particulate matter (P.M.) to
nicotine of examples 1—6, was superior to
that of the control examples 7, 8 and 9 to
a marked degree. The increases in value of
ratio of particulate matter (P.M.) to nicotine
between testings in examples 1 to 6 was not
considered to be significant.

It has been found and it is indicated by
results tabulated in Table 1 that, by the addi-
tion of small quantities of nicotine pectinate or
nicotine alginate an appreciable retention of
nicotine can be secured and transferred to
the smoke whereby the nicotine content of
the smoke is fortified. Furthermore it has
been found that the nicotine content of the
smoke is not appreciably affected by storage
and thus the loss in nicotine has been found
to be no greater than that from the most
stable of the control examples used.

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Table 1

Series 1

TYPE	Nicotine in Material %	P.M.* In Smoke (Mg/Cig.)		Nicotine In Smoke (Mg/Cig.)		Nicotine Transfer %		P.M.* Nicotine Ratio	
		21.5.71.	16.8.71.	21.5.71.	16.8.71.	21.5.71.	16.8.71.	21.5.71.	16.8.71.
Example 1	2.86	30.1	29.0	3.61	3.12	17.1	15.2	8.3	9.3
Example 2	3.90	32.4	30.0	5.02	4.26	17.3	14.7	6.4	7.0
Example 3	2.67	28.2	25.5	3.65	3.06	18.4	15.2	7.7	8.3
Example 4	3.93	29.7	27.1	4.91	4.42	16.8	15.6	6.1	6.1
Example 5	2.78	28.0	26.6	3.62	3.03	17.6	14.8	7.7	8.8
Example 6	4.33	25.4	25.6	5.50	4.79	17.0	14.5	4.6	5.4
Example 7	0.78	17.1	17.2	0.75	0.68	13.0	11.6	27.3	25.3
Example 8	1.44	-	27.4	-	1.67	-	15.6	-	16.4
Example 9	1.93	-	31.4	-	2.16	-	14.4	-	14.5

* Denotes particulate matter free from water and nicotine.

Series 2: Examples 10—20.

Example 10.

5 A sheet of reconstituted tobacco was prepared from a tobacco base consisting of equal proportions of offal and stem (processed mid rib or winnowings).

10 Five pounds (oven dry weight) of winnowings of nicotine content 0.77% were made up to ninety-three pounds with water and the resulting slurry passed through a Probst and Class (P.U.C.) vibration mill at zero setting and then through a Morehouse Industries colloid mill at 0.003 inch interference.

15 Five pounds of offal of nicotine content 1.85% was processed separately by mixing the five pounds (oven dry weight) in sixty-three pounds of water and passing this through a P.U.C. mill at zero setting and then twice through a Manton-Gaulin K3 homogeniser at 3,000p.s.i.

20 The winnowings slurry and the offal slurry were mixed together, and 14.5lbs of Sample 4 nicotine pectinate solution were added.

25 The nicotine fortified slurry was recycled through a P.U.C. mill to achieve thorough mixing and then cast on a Sandvik cast tobacco sheet machine (Sandvik is a Registered Trade Mark). The Sandvik machine was run at 13.5 feet per minute with a 0.030 in casting gate and a 0.5in. slurry head. This produced 11.25lbs of sheet.

30 The nicotine content of the sheet immediately after manufacture was 3.15%. Some of the sheet was stored for eight weeks in open containers under atmospheric conditions of 21° C and 60% RH. The nicotine content after eight weeks was 3.12%, indicating an extremely stable situation.

35 The remainder of the tobacco sheet so prepared was cut and manufactured into filter tipped cigarettes on a Molins Mk. 8 cigarette making machine. The cigarettes were 70mm in length and 25.3mm in circumference and had a dual myria/acetate filter. Twenty-five of the cigarettes were mechanically smoked an an Imperial Tobacco smoking machine to a butt length of 2mm longer than the filter overwrap material using a 35ml, 2 second duration puff one per minute. The smoke condensate collected on Cambridge glass fibre filter pads was analysed for particulate matter and nicotine by weighing the wet deposit on each filter, analysing the deposit for nicotine content by steam distillation (Willits method) and analysing the deposit for water content by gas chromatography. The particulate matter (P.M.) level is thus water and nicotine-free. The results obtained are set out in Table 2.

Example 11

60 A sheet of reconstituted tobacco was prepared according to the method in Example 10 but 14.5lbs of Sample 5 nicotine pectinate solution was used.

The sheet so prepared had a nicotine con-

tent of 2.99% after making and 2.95% after eight weeks storage under condition of 21° C and 60% RH. 65

Filter tipped cigarettes made to the specification outlined in Example 10 gave the results listed in Table 2 when analysed by the standard methods set out in Example 10. 70

Example 12.

A sheet of reconstituted tobacco was prepared in accordance with the method in Example 10 and 14.5lbs of solution Sample 6 nicotine alginate was substituted for the nicotine pectinate solution. 75

The sheet had a nicotine content of 3.00% after casting and 2.97% after eight weeks storage under conditions of 21° C and 60% RH, indicating a very stable product. 80

Filter tipped cigarettes manufactured and analysed according to the methods stated in Example 10 gave the results set out in Table 2. 85

Example 13.

The sheet of example 10 was made up as described in that example. Instead of 14.5lb of Sample 4 nicotine pectinate solution, 12lb of the same sample 4 solution was added to 20lb (oven-dry weight) of sheet mixture whereby the final nicotine content of the cast sheet was 2.08% rather than 3.15%. 90

Again the sheet was made into cigarettes and smoked in the standard way described in example 10 and analysed according to the method of example 10. The results are listed in Table 2. 95

Example 14.

The sheet of example 10 was made up as described in that example, but 12lb of Sample 5 nicotine pectinate solution was added to 20lb (oven-dry weight) of the basic tobacco mixture to provide a nicotine content of 2.12%. 100

The sheet was made into cigarettes, smoked and tested as described in example 10. The results are set out in Table 2. 105

Example 15.

The sheet of example 10 was again prepared but had added thereto 12lb of Sample 6 nicotine alginate solution to 20lb (oven-dry weight) of basic tobacco mixture to provide a final nicotine content of 2.09%. The sheet was made into cigarettes, smoked and analysed as described with reference to example 10. 110

Example 16.

Cut rolled tobacco stem (processed mid rib) was treated with nicotine pectinate to increase its nicotine content to that of commonly used tobacco lamina grades. 120

25lb of the stem containing 12% moisture and 0.56% nicotine was placed in a poly-

these bin and 17½lb of Sample 7 nicotine pectinate solution added in small portions. The mixture was stirred with a paddle to minimise the formation of lumps and to ensure an even application of the nicotine pectinate solution to the stem.

The mixture was allowed to stand overnight and then dried back to 15% moisture in a gas fired rotary drier. The material had a nicotine content of 1.64%.

The material was then manufactured into filter tipped cigarettes on a Molins Mk. 8 cigarette making machine. The cigarettes were 70mm in length and 25.3mm in circumference and had a dual myria/acetate filter. They weighed 1.040g and had a pressure drop of 118m of water.

The cigarettes were smoked using the standard methods previously described in Example 10 and gave the results listed in Table 2 using the analysis technique set out in Example 10.

Example 17.

The objective was to prepare a substitute smoking material in the form of a sheet such that it contained approximately 2% of nicotine and was completely free from any form of tobacco.

To prepare the sheet material, a solution of 35g of slow-set citrus pectin (obtained from H.P. Bulmers Ltd.) in 700 ml of water was prepared with the aid of a Silverson mixer. The slow-set citrus pectin had a degree of esterification of 63% and an acidity of 1.6 milli-equivalents per gramme. A second mixture was prepared by dispersing 30g calcium carbonate, 5g potassium carbonate, 5g celite (Hyflo Super Cel grade), 5g magnesium hydrogen phosphate and 2g titanium dioxide in 250ml of water. To this suspension was added 6g of citric acid.

This slurry of inorganic materials was added to the citrus pectin solution and thoroughly mixed with the Silverson stirrer. A third solution of 9g of Sample 2 nicotine pectinate in 250ml of water was then added, followed by 5g of glycerol. The resulting slurry was thoroughly mixed again, and then passed through a Premier 84 colloid mill adjusted to a running clearance of 0.003in. to remove any lumps.

The slurry was cast on a continuous stainless steel band using an 1.1mm casting gate and dried using Infra Red heating lamps. The dried sheet was moistened to facilitate removal and doctored off the band.

The prepared sheet was cut at 40c.p.i. using a Hauni sample cutting machine, Model TSH V and manufactured into filter tipped cigarettes using an Efka-Werke "Privileg" hand machine. These cigarettes were 83mm long, 25mm in circumference and contained 1.0g of filler material. The filler material contained 1.93% nicotine. The filter was a mono acetate type.

When smoked and analysed by the standard methods described in Example 10 the results listed in Table 2 were obtained.

Example 18.

To provide a control against which the effects of nicotine fortification can be compared, a sample of commercially available low tar yielding sheet containing 0.5% of nicotine was cut and manufactured into filter tipped cigarettes in the manner described in Example 10. The cigarettes were 70mm in length and 25.3mm in circumference and contained 0.70g of sheet.

When smoked under the standard conditions of Example 10 and analysed as described in Example 10, the Table 2 results were obtained.

Example 19.

To provide a standard against which the examples 10—18 could be compared, a standard filter tip cigarette, commercially available in the United Kingdom and being of the same specification as the cigarettes referred to in Example 10 were smoked and analysed by the standard methods set out in Example 10. The nicotine content of the filler in the cigarette was determined before smoking as 2.01%. The smoke analysis is given in Table 2.

Example 20.

To demonstrate the effectiveness of nicotine pectinate as a means of increasing the nicotine content of stem (processed mid rib), a control sample of untreated stem containing 0.66% nicotine was manufactured into filter tipped cigarettes in the manner described in Example 16 and with the same physical dimensions as the cigarettes in Example 16. When smoked and analysed using the standard methods described in Example 10 the results set out in Table 2 were obtained.

Table 2.

Referring to Table 2 it will be noted that the additive is extremely stable. In all cases only a small loss of nicotine derivative of pectinic or alginic acid was noted in the eight weeks following application to the base tobacco material. This is far superior to previous attempts to find a stable nicotine fortification material.

Regarding the nicotine transfer figures, it will be seen that these are extremely good for all examples which were fortified. Over 16% of the nicotine in each starting material was transferred to the smoke and presented to the filter. It will be seen that this compares favourably with the standard or control cigarette results. Thus the commercially available cigarette tested produced a transfer figure of 21.2%. The test results indicate

that very little reduction in this figure is to be expected even with fortified materials and blends.

5 It will be appreciated that the materials tested were all basic materials in Examples 10—17 i.e. no blends of materials were used. However, such blends could of course be used in any commercial use of the material. This should not affect the nicotine transfer from the mixture or the individual materials.

10 The ratio of particulate matter (P.M.) to nicotine is seen to be extremely good in the Examples 10—16, being far lower than in the control Examples 18 and 20 and being of the same order as the commercial cigarette

15 Example 19 for the same initial nicotine con-

tent of approximately 2% (compare Examples 13—15 with Example 19).

The results indicate that nicotine derivatives of alginic and pectinic acids provide extremely stable nicotine fortification agents which are effective in efficiently transferring an acceptable percentage of the nicotine to the smoker. Furthermore the fortification agent is useful in fortifying acceptable smoking base materials such as not only natural lamina tobacco but also stem (mid rib), reconstituted tobacco sheet and synthetic smoking materials. Particularly with respect to the last two materials, it should be noted that best results are obtained if the pH of the material is kept below approximately 6.5.

Table 2

Series 2

TYPE	Nicotine content of material %		Mean no. of puffs per cig.	P.M.* in smoke (mg/cig.)	Nicotine in smoke (mg/cig.)	Filter Retention %	Nicotine Transfer %	P.M. Nicotine Ratio
	When made	After 8 weeks						
Example 10	3.15	3.12	8.7	17.0	1.53	54.1	16.6	11.1
Example 11	2.99	2.95	8.1	14.2	1.41	54.6	18.6	10.1
Example 12	3.00	2.97	8.4	17.3	1.56	53.0	18.6	11.1
Example 13	2.08	2.06	7.0	14.2	0.98	53.7	18.5	14.5
Example 14	2.12	2.09	7.7	17.1	1.08	55.9	18.7	15.8
Example 15	2.09	2.06	7.6	15.8	1.14	53.0	20.3	13.9
Example 16	1.64	-	8.2	10.0	0.71	56.1	15.1	14.1
Example 17	1.93	-	8.7	12.3	1.70	29.0	17.0	7.2
Example 18	0.50	-	8.0	9.1	0.30	52.0	19.0	33.0
Example 19	2.01	-	11.6	21.0	1.49	47.8	21.2	14.1
Example 20	0.66	-	8.4	11.4	0.29	54.2	14.6	39.4

* Denotes particulate matter free from water and nicotine.

Furthermore Curie point pyrolysis followed by gas/liquid chromatography tests on the nicotine pectinate and nicotine alginate employed indicated that maximum yield of nicotine occurs between 500 and 550° C i.e. at the temperature immediately behind the burning coal of a cigarette. It would appear from the results obtained that nicotine derivatives of pectinic and alginic acids offer substantially attractive additives to tobacco products providing a nicotine agent which will fortify the nicotine content of the product whilst remaining relatively stable over a period of months and yielding the nicotine at the optimum temperature of 500 to 550° C and yet being a relatively acceptable and harmless ingredient making it a most suitable and attractive additive.

Pectins, particularly those from tobacco plants, are well known as binders or sheet strengtheners in the manufacture of reconstituted tobacco sheet or synthetic smoking materials and thus the addition of nicotine pectinate has the added attraction that the pectinate so added assists in binding and strengthening reconstituted or synthetic sheet. Thus not only is the sheet fortified with nicotine but also a strengthening agent is added without the need to add additional substances as is done in reconstituted or synthetic sheet manufacture.

With the present invention there is provided a stable and effective nicotine fortification material being the nicotine derivatives of a polyuronic acid.

Although all examples have used nicotine derivatives of polygalacturonic and polymannuronic acids only, it is considered that any polyuronic acid can be used in view of the closely similar properties of these acids. Thus nicotine derivatives of polyglucuronic acid and of polyguluronic acid would be useful nicotine fortification agents but, like all polyuronic acids other than polygalacturonic and polymannuronic they are not readily available in bulk and thus at this time are not commercially attractive. However this does not detract from their effectiveness. The examples have been limited to the two readily available and commercially attractive poly-

uronic acid derivatives but the invention embraces the nicotine derivatives of all polyuronic acids.

WHAT WE CLAIM IS:—

1. A process for making a smoking material comprising adding to a smokable base material a nicotine derivative of a polyuronic acid, whereby on pyrolysis of the resulting smoking material controlled amounts of nicotine are released into the smoke. 55
2. A process as claimed in claim 1 wherein the polyuronic acid is a polygalacturonic acid or polymannuronic acid. 60
3. A process as claimed in claim 2 wherein the polygalacturonic acid is pectinic acid and the derivative is nicotine pectinate. 65
4. A process as claimed in claims 3 wherein the pectinic acid has a degree of esterification of the order of 30—35% and an acidity of 1.5 to 3.5 milli-equivalents per gram. 70
5. A process as claimed in claim 4 wherein the acidity is 2 to 3.5 milli-equivalents per gram.
6. A process as claimed in claim 2 wherein the polymannuronic acid is alginic acid and the derivative is nicotine alginate. 75
7. A process as claimed in any preceding claim wherein the base material is tobacco lamina, stem (mid rib), reconstituted sheet, or synthetic smoking material which is tobacco free. 80
8. A process as claimed in any preceding claim wherein the amount of derivative added to the base material is 3% to 13% by weight.
9. A process for making a smoking material containing reconstituted tobacco sheet, substantially as described with reference to any one of Examples 1 to 4 and 10 to 14. 85
10. A process for making a smoking material containing cut rolled tobacco stem, substantially as described with reference to Example 16. 90
11. A process for making a smoking material containing no tobacco, substantially as described with reference to Example 17. 95

B. D. FREEMAN,
Chartered Patent Agent,
Agent for the Applicants.

Furthermore Curie point pyrolysis followed by gas/liquid chromatography tests on the nicotine pectinate and nicotine alginate employed indicated that maximum yield of nicotine occurs between 500 and 550° C i.e. at the temperature immediately behind the burning coal of a cigarette. It would appear from the results obtained that nicotine derivatives of pectinic and alginic acids offer substantially attractive additives to tobacco products providing a nicotine agent which will fortify the nicotine content of the product whilst remaining relatively stable over a period of months and yielding the nicotine at the optimum temperature of 500 to 550° C and yet being a relatively acceptable and harmless ingredient making it a most suitable and attractive additive.

Pectins, particularly those from tobacco plants, are well known as binders or sheet strengtheners in the manufacture of reconstituted tobacco sheet or synthetic smoking materials and thus the addition of nicotine pectinate has the added attraction that the pectinate so added assists in binding and strengthening reconstituted or synthetic sheet. Thus not only is the sheet fortified with nicotine but also a strengthening agent is added without the need to add additional substances as is done in reconstituted or synthetic sheet manufacture.

With the present invention there is provided a stable and effective nicotine fortification material being the nicotine derivatives of a polyuronic acid.

Although all examples have used nicotine derivatives of polygalacturonic and polymannuronic acids only, it is considered that any polyuronic acid can be used in view of the closely similar properties of these acids. Thus nicotine derivatives of polyglucuronic acid and of polyguluronic acid would be useful nicotine fortification agents but, like all polyuronic acids other than polygalacturonic and polymannuronic they are not readily available in bulk and thus at this time are not commercially attractive. However this does not detract from their effectiveness. The examples have been limited to the two readily available and commercially attractive poly-

uronic acid derivatives but the invention embraces the nicotine derivatives of all polyuronic acids.

WHAT WE CLAIM IS:—

1. A process for making a smoking material comprising adding to a smokable base material a nicotine derivative of a polyuronic acid, whereby on pyrolysis of the resulting smoking material controlled amounts of nicotine are released into the smoke.

2. A process as claimed in claim 1 wherein the polyuronic acid is a polygalacturonic acid or polymannuronic acid.

3. A process as claimed in claim 2 wherein the polygalacturonic acid is pectinic acid and the derivative is nicotine pectinate.

4. A process as claimed in claims 3 wherein the pectinic acid has a degree of esterification of the order of 30—35% and an acidity of 1.5 to 3.5 milli-equivalents per gram.

5. A process as claimed in claim 4 wherein the acidity is 2 to 3.5 milli-equivalents per gram.

6. A process as claimed in claim 2 wherein the polymannuronic acid is alginic acid and the derivative is nicotine alginate.

7. A process as claimed in any preceding claim wherein the base material is tobacco lamina, stem (mid rib), reconstituted sheet, or synthetic smoking material which is tobacco free.

8. A process as claimed in any preceding claim wherein the amount of derivative added to the base material is 3% to 13% by weight.

9. A process for making a smoking material containing reconstituted tobacco sheet, substantially as described with reference to any one of Examples 1 to 4 and 10 to 14.

10. A process for making a smoking material containing cut rolled tobacco stem, substantially as described with reference to Example 16.

11. A process for making a smoking material containing no tobacco, substantially as described with reference to Example 17.

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