

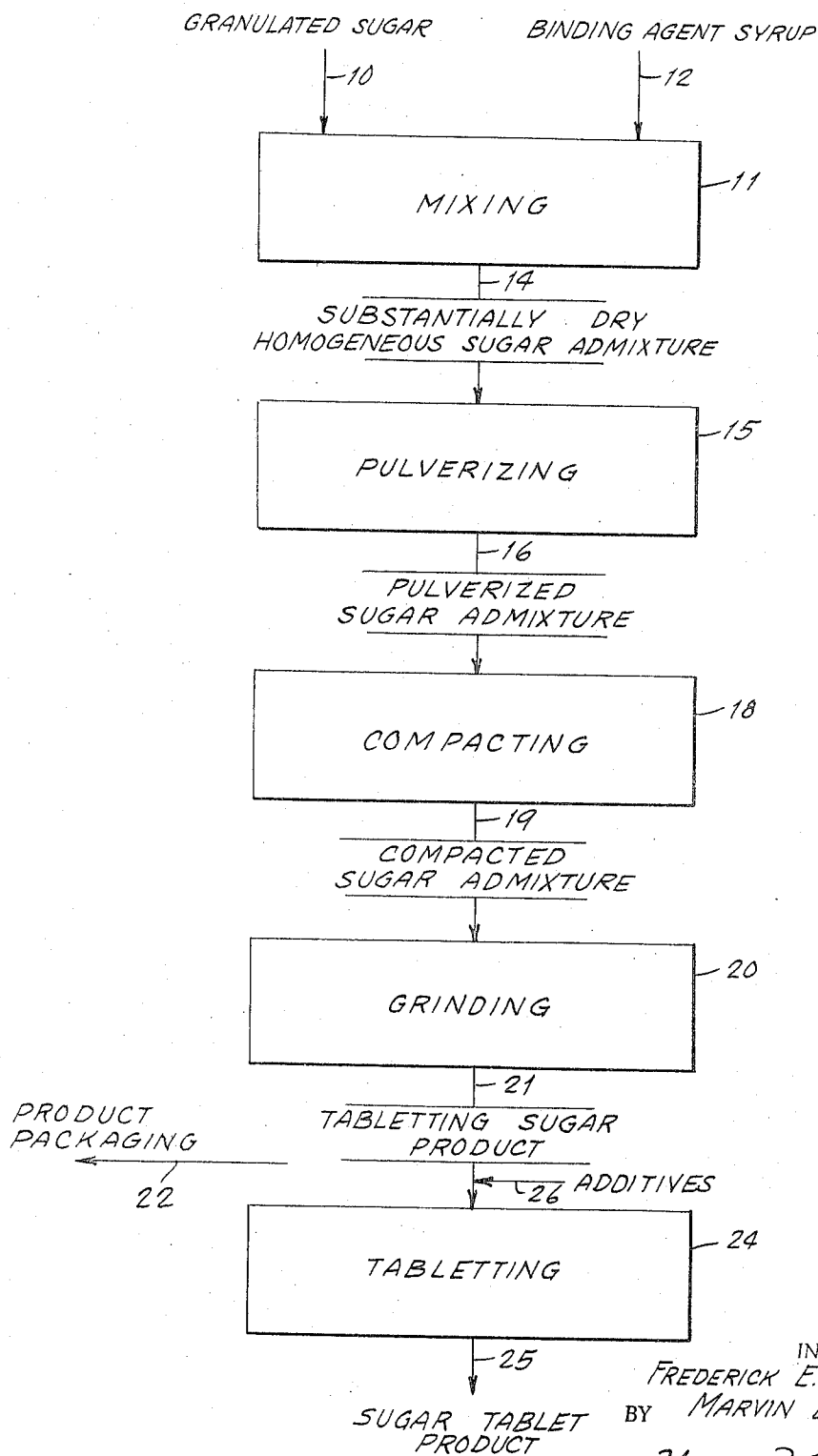
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F. E. REIMERS ETAL

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TABLETTING SUGAR AND METHOD OF PREPARING SAME

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INVENTORS
FREDERICK E. REIMERS
BY MARVIN D. MILLER

Thomas J. Moran
ATTORNEY

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TABLETTE SUGAR AND METHOD OF PREPARING SAME

Frederick E. Reimers, Westfield, N.J., and Marvin D. Miller, Oceanside, N.Y., assignors to American Sugar Company, New York, N.Y., a corporation of New Jersey

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This invention relates to sugar products. More particularly, this invention relates to tableting sugar and method of preparing same. More particularly, this invention relates to a sugar product useful in the direct compaction of sugar-containing pharmaceutical tablets such as lozenges, confectionery pressed tablets, and the like.

At present the use of sugar, such as sucrose, in granulated or powdered form as a carrier or excipient for drugs and the like in the manufacture of pressed tablets is limited to the so-called wet granulation procedure or the so-called dry slugging procedure. Granulated sucrose by itself cannot be compacted in a tablet press since the crystals of granulated sucrose when subjected to tableting pressure do not flow and do not have the elasticity to cause their realignment into adjacent areas where they can bind together to form a satisfactory pressed tablet. Further, granulated sugar when tabletted does not produce the desired smoothness and textural mouth feel required of lozenges and confectionery.

The operations of wet granulation or dry slugging in a sugar tableting operation are cumbersome and lengthy, particularly when compared to direct compaction where blending and compaction or compression alone are employed in the manufacture of pressed tablets.

The wet granulation method usually requires the sequence of operations (a) blending excipient and active materials, (b) preparation of binder solution, such as sugar syrup, gelatin solution, glucose solution, etc., (c) mixing these materials, (d) passing the mixed materials through a coarse screen or extruding the mixed materials therethrough, (e) distributing the material in trays and placing the same in drying ovens to remove excess moisture, (f) regranulating and screening to a desired particle-size, (g) blending with lubricant, and (h) compressing in a tablet press.

The dry slugging method usually requires the sequence of operations: (a) blending excipient and active materials, (b) slugging or precompressing into a large tablet or slug, (c) grinding of slugs, (d) screening out fines and reslugging same, (e) blending with lubricant and compressing in tablet press.

Also, as indicated hereinabove, powdered sugar by itself cannot be used in the preparation of tablets by direct compaction. Powdered sugar does not flow properly and tends to hang-up in the feed hopper of the tableting press. Induced or forced-flow feeders are of some help in overcoming these problems but satisfactory results are not always obtainable. Further, tablets made of directly compacted powdered sugar do not have the cohesive properties required of a good, firm tablet and necessary for satisfactory handling in tablet packaging equipment. Also tablets produced by the direct compaction of powdered sugar are subject to binding in the press, sticking and picking, capping and laminating and chipping and granulating.

It is an object of this invention to provide a new sugar product particularly useful as a tableting sugar.

It is another object of this invention to provide a sugar product having satisfactory cohesive properties and particularly useful as a tableting sugar product.

It is a further object of this invention to provide a process for the manufacture of tableting sugar.

Still another object of this invention is to provide an improved sugar tablet.

Still another object of this invention is to provide a method of producing tableting sugar having satisfactory anti-caking and flow properties and suitable for use in the manufacture of tableting sugar containing moisture-sensitive materials such as medicinals, flavor agents and the like.

Yet another object of this invention is to provide a method of manufacturing tableting sugar containing in association therewith during manufacture a heat sensitive material.

How these and other objects of this invention are accomplished will become apparent in the light of the accompanying disclosure and with reference to the accompanying drawing which schematically illustrates various steps in accordance with this invention for the production of tableting sugar.

In accordance with this invention a method of producing tableting sugar, and an improved tableting sugar, as well as improved sugar tablets, are provided by blending under vigorous mixing conditions an admixture comprising binding agent and sugar to yield a substantially dry, homogeneous sugar admixture. The substantially dry, homogeneous sugar admixture is then subjected to grinding, compacting and a further grinding operation in the aforesaid sequence of operations to yield a sugar product useful in the manufacture of sugar tablets.

More particularly, in accordance with one embodiment of this invention for the preparation of tableting sugar, a concentrated invert sugar syrup, or a concentrated corn syrup, containing about 95% by weight solids at an elevated temperature of about 135° C., is admixed with granulated sucrose under mixing conditions of turbulent, shear and impact action so as to produce a resulting substantially dry, homogeneous admixture in a form suitable for immediate grinding to a powdered sugar fineness in ordinary sugar grinding equipment. The amount of concentrated syrup may comprise from about 1 to about 25%, or more, by weight of the material undergoing admixture.

Following the mixing operation carried out under conditions of turbulent, shear and impact action to produce a substantially dry, homogeneous admixture, the resulting admixture is ground to a desired fineness or particle-size, e.g., a particle-size such that approximately 95% of the ground admixture passes through a 200 mesh Tyler screen. This particular particle-size has been found to be particularly useful but finer and coarser particle-sizes or grinds may be employed depending upon the mouth, feel and textural properties desired in the finished, pressed sugar tablet.

This ground sugar admixture exhibits poor flow properties and in order to impart thereto the desired flow properties it has been found necessary in accordance with the practice of this invention to subject the ground sugar admixture to compacting. The resulting compacted sugar, such as in the form of flakes or ribbons, is then granulated, ground or subjected to comminution, such as in a hammermill, to a size having suitable flow and anti-caking properties. Ground compacted sugar products having the following particle-size analyses have been found to provide good flow properties and to be useful as tableting sugars:

Analysis A

Particle-size, Tyler mesh:	Percent by wt.
On 14	2
On 26	40
On 35	15
On 48	13
On 100	18
Through 100	12

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Analysis B

Particle-size, Tyler mesh:	Percent by wt.
On 28 -----	9
On 48 -----	40
On 60 -----	14
On 100 -----	25
On 150 -----	6
On 200 -----	3
Through 200 -----	3

The analyses A and B are typical and other analyses are useful in the practice of this invention. It is desirable, however, that the particle size analyses be such as to impart good flow properties to the tableting sugar and textural appearance in the tabletted product.

The thus-prepared sugar product is suitable for feeding to a tableting press, desirably admixed with about 1% by weight of a suitable lubricant, such as magnesium stearate, to provide good, firm tablets.

Tableting sugar having the particle-size of Analysis A and containing 5% by weight invert sugar, 1% magnesium stearate, the remainder comprising essentially only sucrose, was evaluated on a Colton Rotary Press at a tablet production rate of 1035 tablets per minute. The following test results were observed:

Flowability -----	Excellent.
Compressibility -----	Good.
Color -----	Satisfactory.
Sheen -----	Good.
Average weight -----	365 mg.
Hardness (Stokes) -----	7.1 kg./in. sq.
Disintegration time -----	5 min. and 5 sec.
Friability -----	0.35%.

During the tableting operation no capping, laminating or sticking to the punches was observed. Although tableting sugar prepared in accordance with this invention and containing 5% by weight invert sugar has been found to be particularly useful, for certain applications, however, in which greater binding or cohesiveness is required or desired, larger amounts, such as up to about 20% by weight invert or other effective binding agent material, such as corn syrup, may be included in the tableting sugar product.

Other tableting tests employing tableting sugar prepared in accordance with this invention and containing different amounts of invert (reducing sugars) as binding agent were carried out with a Colton Rotary Press at a tablet production rate of 1035 tablets per minute employing 1% by weight magnesium stearate in the tableting sugar as lubricant. The following test results were observed:

	3% Invert Content	7% Invert Content
Flowability -----	Excellent.	Excellent.
Compressibility -----	Fair to good.	Fair.
Color -----	Satisfactory.	Satisfactory.
Sheen -----	Fair to good.	Fair.
Average Weight -----	366 mg.	364 mg.
Hardness (Stokes) -----	6.3 kg./in. sq.	9.3 kg./in. sq.
Disintegration Time -----	5 min. and 15 sec.	4 min. and 25 sec.
Friability -----	3.3%.	0.3%.
	15 tablets capped out of 4,000 and a substantial amount of edge chipping during the friability test.	No capping, laminating or sticking to punch faces observed during test.

Additional tests were carried out wherein corn syrup was employed as the binding agent in the manufacture of tableting sugar in accordance with this invention. In these tests a commercial corn syrup of 42 DE and approximately 81% solids was concentrated to approximately 90% solids and admixed and dispersed on granulated

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sugar in an amount of about 5% of the resulting admixture. The admixture was pulverized, compacted and ground as described hereinabove and the final ground material was tabletted in a Colton Rotary Press at a rate of 1035 tablets per minute employing 1% magnesium stearate as a lubricant. The following test results were observed:

Flowability -----	Excellent.
Compressibility -----	Excellent.
Color -----	Satisfactory.
Sheen -----	Good.
Hardness (Stokes) -----	8.25 kg./in. sq.
Disintegration time -----	4 min. and 9 sec.
Average weight -----	357 mg.
Friability -----	Negligible.

No capping, laminating or sticking to punch faces were observed.

In another test, a dry mixture of glucose and dextrins (corn syrup solids) was thoroughly admixed with granulated sugar, pulverized to a fineness so that 90% passed through a 200 mesh Tyler screen, compacted and reground as described hereinabove to yield a particle-size range substantially as Analysis A. Test materials so prepared and containing 3% corn syrup solids and 10% corn syrup solids were tabletted in a Colton Rotary Press at a rate of 1035 tablets per minute using 1% magnesium stearate as a lubricant. In each of the tests, although flowability, color and sheen were satisfactory, the binding properties of the added dry corn syrup solids were poor, resulting in capping and unsatisfactory tablet hardness. The test material containing 3% corn syrup solids produced a tablet having a hardness of approximately 2 kg./in. sq. and the 10% corn syrup solids test material gave a tablet having a hardness of about 4 kg./in. sq. These tests indicated that the use of the binding agent in dry, solid form did not yield a satisfactory tableting sugar.

It was observed, however, in still other tests that the addition of moisture to a dry blend of granulated sugar and binding agent, such as produced by thoroughly admixing sucrose and corn syrup solids, produced a satisfactory tableting sugar. However, from a practical point of view the addition of moisture to the dry solids binding agent-sugar mixture is not completely satisfactory since the resulting mixture exhibits reduced flowability and therefore results in unsatisfactory weight and size control in the finished tablets. Also, mottling, due to the inability to evenly wet all the particles in the mixture, was observed in the finished tablets.

Heat sensitive and/or moisture sensitive active materials, such as medicinals, flavoring agents, coloring agents and the like to be incorporated in the finished tablets may be added at substantially any time during processing in accordance with this invention, such as during mixing, pulverizing and grinding.

Referring now to the drawing which schematically illustrates the process flow in accordance with this invention, granulated sugar, such as granulated sucrose, is supplied at about room temperature via line 10 to mixing unit 11. Hot, about 135° C. concentrated binding agent syrup, such as a concentrated invert syrup having solids content of above about 90% or a concentrated corn syrup of substantially the same solids content, is supplied to mixing unit 11 via line 12. Within mixing unit 11 the granulated sucrose and binding agent syrup are thoroughly dispersed and homogeneously admixed under turbulent, shear and impact action to produce a substantially dry, homogeneous sugar admixture having a moisture content less than about 1%.

A suitable mixer for mixing unit 11 is a mixer manufactured and sold by The Strong-Scott Manufacturing Company of Minneapolis, Minn., and known as the Turbulizer. The Turbulizer in mixing and dispersing combines turbulent, shear and impact actions, sometimes re-

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ferred to as turbulizing action. In construction the Turbulizer employs a shell within which is provided a high speed rotating shaft to which is fitted a plurality of paddles, the pitch of which is adjustable, as well as their peripheral speed and the distance separating the paddles and the shell. Material to be dispersed and mixed is supplied at one end of the shell and discharged at the other end. If desired the shell and/or shaft may be jacketed for heat exchange.

Following mixing or turbulizing, the resulting substantially dry, homogeneous sugar admixture is supplied via line 14 to pulverizing unit 15. Pulverizing unit 15 may be any suitable means, such as a hammermill, e.g. a Mikro-Mill. Following the pulverizing operation the pulverized sugar admixture is supplied via line 16 to compacting unit 18. A Fitzpatrick Chilsonator and an Allis-Chalmers compactor have been found to be satisfactory to effect compacting of the pulverized sugar. The Chilsonator compacts the sugar admixture by passing the sugar admixture through a set of serrated rolls which can exert pressures up to 120,000 pounds against each other. The resulting compacted sugar issues from the compacting or so-called Chilsonating operation in the form of compacted flakes or ribbons and is supplied via line 19 to grinding unit 20 for regrounding. A Fitzpatrick comminuting mill or a Stokes "Tornado" comminuting mill is satisfactory for effecting grinding of the compacted sugar. The resulting reground compacted sugar is supplied via lines 21 and 22 to a product packaging operation as finished tableting sugar product or, if desired, the tableting sugar product may be supplied directly to a tableting unit 24 for the production of sugar tablets recovered as product via line 25.

If desired, various additive materials may be added with suitable blending to the tableting sugar, as indicated via line 26. Such additives might include pharmaceuticals, medicinals, coloring agents, flavoring agents or other active ingredients. If desired, depending upon the physical properties of the active additive materials, these materials may be added to the process during mixing 11, pulverizing 15 and/or grinding 20. A lubricant such as a stearate, e.g. magnesium stearate, is desirably added to the tableting sugar just prior to tableting.

The following examples are illustrative of the practice of this invention:

Example No. I

A commercial sugar syrup sold by the American Sugar Co. as Crystal 50, containing 53% invert (dry basis) and testing 74.2° Brix was heated to 233° F. in a gas fired open kettle to 92.4° Brix. This material was fed to a Turbulizer at a rate of 400 cc. of invert sugar concentrate per 4540 grams of fine granulated sucrose per minute. The Turbulizer was run at low speed (900 r.p.m.). The mixture of granulated sucrose and invert syrup exciting the Turbulizer was slightly tacky and had an invert content of 4.9%. The sucrose invert mixture was ground in a Mikro Pulverizer using a 0.027" screen. The material had a screen analysis of 91.6% through a Tyler 200 mesh screen. The ground sugar which was essentially dry had a moisture content of 0.78%. The ground sugar was fed to a Fitzpatrick Chilsonator. In the Chilsonator the ground sugar compacted into large, firm flakes which were subsequently sized in a Fitzpatrick Comminuting Mill operated with a 12 mesh screen and at a rotor speed of 1750 r.p.m.

The screen analysis of the sugar product was:

Tyler mesh:	Percent
+14	1.2
+28	26.7
+35	11.2
+48	10.4
+100	21.3
-100	29.2

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Screening of the sugar product on a Sweco Sifter equipped with a 100 mesh screen resulted in a finished sugar product of the following analysis:

Tyler mesh:	Percent
+14	1.4
+28	40.7
+35	16.9
+48	13.9
+100	19.5
-100	7.6

This material flowed well and produced good firm tablets without any difficulty.

Example No. II

Granulated sucrose and a concentrated invert sugar syrup produced by heating Crystal 50 syrup in an open gas fired kettle to 135° C. to a solids content of about 93.6% by weight, invert sugar content of 7.32%, were turbulized. The resulting substantially dry homogeneous sugar mixture had a moisture content in the range 0.26-0.35% and an invert sugar content in the range about 4.6 to about 5.5% by weight.

The turbulized sugar mixture appeared uniform and was in a condition which permitted grinding without any difficulty.

This turbulized sugar mixture was ground to 90%-200 mesh and compacted in a Chilsonator.

The mixture was compacted into a good firm flakes admixed with some fines. Analysis of the compacted sugar mixture showed 87.2% to be larger than 9 mesh Tyler and 12.8% to be smaller than a 9 mesh Tyler.

The compacted sugar mixture was then sized in a Fitzpatrick Comminuting machine at two different settings:

(a) Using a 12 mesh screen and a rotor speed of 1680 r.p.m. a product was obtained analyzing:

Tyler mesh:	Percent
+14	1.7
+28	39.7
+35	12.8
+48	11.2
+100	20.7
-100	13.9

(b) Using a #12 screen and a rotor speed of 4300 r.p.m. a product was obtained analyzing:

Tyler mesh:	Percent
+28	8.2
+48	40.0
+60	10.5
+100	25.2
+150	5.5
+200	5.6
-200	5.0

Both materials showed good flowability and made good firm tablets.

Tablet weights were determined to indicate weight variation between individual tablets and all tests indicated variations within U.S. Pharmacopoeia Standards.

Example No. III

Corn syrup of 43° Baumé and 42 DE was heated to 126° C. in an open gas fired kettle to reach a solids content of approximately 87%. This material was incorporated by turbulizing onto granulated sucrose at a feed ratio of 0.6 lb. of syrup per 10 lbs. of granulated sucrose to yield 5% corn syrup solids in the resulting turbulized sugar mixture. The mixture had a moisture content of approximately 1.2% by weight.

The mixture was ground on a Mikro Pulverizer to 90%-200 mesh Tyler and compacted in a Chilsonator.

The compacted sugar was ground in a Fitzpatrick mill using a 12 mesh screen and a rotor speed of 1750 r.p.m., and the resulting comminuted sugar was screened on a

Sweco Sifter equipped with a 100 mesh screen to remove some fines. The screened sugar had the following screen analysis:

Tyler mesh:	Percent
+14 -----	1.4
+28 -----	40.7
+35 -----	16.9
+48 -----	13.9
+100 -----	19.5
-100 -----	7.6

This sugar product showed good flowability and produced good tablets on a Colton Rotary Press.

By following the practice of this invention it is possible to prepare a tableting sugar in essentially dry form but having the cohesive properties of tableting sugar wherein wet binders, i.e. with binding agents having a moisture content substantially greater than 5%, are employed. Further, in accordance with the practice of this invention, it is possible to incorporate an essentially sticky, hygroscopic material, such as invert or corn syrups, in such a manner that this material does not interfere with the flowability of the tableting sugar, thereby providing good tablet weight control without exhibiting die sticking or punch face sticking. Also, in the preparation of tableting sugar in accordance with this invention the addition of active ingredients in an amount from about 1 to about 90% in the finished tablet, depending upon the character of the ingredients, is possible. Special ingredients, such as phenobarbital can readily be provided in a tableting sugar prepared in accordance with this invention to yield sugar tablets containing as high as 40% by weight phenobarbital.

It is possible in the practice of this invention to add coloring agents, e.g. lake or soluble colors, to the mixture of sugar and binding agent prior to grinding or pulverizing and to prepare a material after compacting and sizing to a desired size which has good coloring properties and which can be used to produce uniformly colored tablets. It has also been founded that the tableting sugar products, such as the pulverized material issuing from the pulverizing operation 15 via line 16, can be admixed with from about 3 to about 5% by weight corn starch or similar material to impart improved anti-caking properties.

The tableting sugar product of this invention, although particularly useful in the manufacture of sugar tablets, also exhibits other special utility. This invention provides a method of adding varying amounts of invert sugar to sugar such as granulated sucrose in a manner permitting the resulting admixture of invert and sugar to be ground to substantially any desired fine particle-size, such as 4 $\times$, 6 $\times$ or 10 $\times$. An icing prepared from such fine ground sugars exhibits greater resistance to drying-out and cracking, thereby making it possible to maintain a soft, fresh, slow-crystallizing icing for longer periods of time.

Although emphasis has been placed in this disclosure on the use of invert syrup and corn syrup as the binding agent in the preparation of tableting sugars in accordance with this invention, other binding agents in syrup form, such as dextrin, alginates, carboxymethylcellulose (CMC) vegetable gums and mixtures thereof or in combination with invert syrup and corn syrup are useful in the practice of this invention. Further, other sugars such as dextrose, lactose, maltose, etc. in granulated or powdered form in addition to sucrose can be employed in the practice of this invention to produce a tableting sugar.

As will be apparent to those skilled in the art in the light of the foregoing disclosure, many modifications, substitutions and alterations are possible in the practice of this invention without departing from the spirit or scope thereof.

We claim:

1. A sugar product suitable for tableting consisting essentially of a major amount of granulated sugar and a minor amount of a binding agent, said sugar product

having been prepared by subjecting an admixture containing said granulated sugar and said binding agent in the form of a concentrated syrup containing above about 87% by weight of binding agent to mixing to produce a substantially dry, homogeneous sugar admixture containing less than about 1% by weight moisture, subjecting the resulting sugar admixture to grinding to produce a ground sugar admixture having a particle size such that at least about 90% by weight passes through a 200 mesh Tyler sieve, compacting the resulting ground sugar admixture and subjecting the resulting compacted sugar admixture to a further comminution operation.

2. A sugar product in accordance with claim 1 wherein said binding agent is selected from the group consisting of invert sugar, corn syrup solids, dextrin, alginates, water soluble vegetable gum and carboxymethylcellulose.

3. A sugar product in accordance with claim 1 wherein said sugar is selected from the group consisting of sucrose, dextrose, lactose and maltose.

4. A sugar product in accordance with claim 1 having the following particle-size analysis:

Particle-size, Tyler mesh:	Percent by weight
On 14 -----	2
On 26 -----	40
On 35 -----	15
On 45 -----	13
On 100 -----	18
Through 100 -----	12

5. A sugar product in accordance with claim 1 having the following particle-size analysis:

Particle-size, Tyler mesh:	Percent by weight
On 28 -----	9
On 48 -----	40
On 60 -----	14
On 100 -----	25
On 150 -----	6
On 200 -----	3
Through 200 -----	3

6. A sugar product comprising the product of claim 1 in the form of a pressed sugar tablet.

7. A sugar product in accordance with claim 1 consisting essentially of about 1-20% by weight of said binding agent, the remainder of said product being substantially sucrose.

8. A sugar product in accordance with claim 1 consisting essentially of about 3-7% by weight invert sugar, the remainder of said product being substantially sucrose.

9. A sugar product in accordance with claim 1 consisting essentially of about 5% corn syrup solids, the remainder of said product being substantially sucrose.

10. A method of producing tableting sugar comprising subjecting an admixture of granulated sucrose and a concentrated syrup containing above about 87% by weight binding agent dissolved therein to vigorous mixing under conditions to produce a substantially dry, homogeneous sugar admixture containing less than about 1% by weight moisture, subjecting said sugar admixture to pulverizing to produce a powdered, homogeneous sugar admixture having a particle size such that at least about 90% by weight passes through a 200 mesh Tyler sieve, compacting the resulting pulverized sugar admixture and comminuting the resulting compacted sugar admixture to a desired particle-size.

11. A method in accordance with claim 10 wherein said concentrated syrup is a syrup containing about 95% by weight invert sugar.

12. A method in accordance with claim 10 wherein said concentrated syrup contains about 95% by weight corn syrup solids.

13. A method in accordance with claim 10 wherein said powdered, homogeneous sugar admixture is compacted by passing said admixture through a set of pressure rolls.

14. A method of producing sugar tablets comprising
 subjecting an admixture of granulated sucrose and a
 binding agent in concentrated syrup form containing
 above about 87% by weight binding agent to vigorous
 mixing under conditions to produce a substantially dry,
 homogeneous sugar admixture having a moisture content
 not more than about 1%, subjecting said sugar admixture
 to pulverizing to produce a powdered, homogeneous sugar
 admixture, compacting the resulting pulverized sugar ad-
 mixture having a particle size such that at least about
 90% by weight passes through a 200 mesh Tyler sieve,
 comminuting the resulting compacted sugar admixture
 to a desired particle-size and pressing the resulting
 ground sugar admixture into a mold to produce a sugar
 tablet of a desired configuration.

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ALBERT T. MEYERS, *Primary Examiner*.

JULIAN S. LEVITT, *Examiner*.

G. A. MENTIS, *Assistant Examiner*.