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		TENT DECENIED ATED CELLUL OSE SALISAGE CASING

(54) Title: THIN-WALLED, LOW PLASTICIZER CONTENT REGENERATED CELLULOSE SAUSAGE CASING

(57) Abstract

A regenerated cellulose sausage casing which has a basis weight, wall thickness and plasticizer content substantially lower than that of casing products produced in the normal manner by the viscose process but has sufficient strength and flexibility characteristics to undergo high speed commercial shirring and meat stuffing operations with a minimum of breakage and pinholing.

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THIN-WALLED, LOW PLASTICIZER CONTENT REGENERATED CELLULOSE SAUSAGE CASING

BACKGROUND OF THE INVENTION

Field of the Invention:

This invention relates to a regenerated cellulose sausage casing having a reduced cellulose and plasticizer content and a process for preparing the casing.

Prior Art

Artificial sausage casings made entirely of regenerated cellulose have been widely used in the processing of frankfurters and related sausage products for a number of years. The basic process for the manufacture of small diameter casings of regenerated cellulose is known as the "viscose process", as for example described in U.S. 2,999,756 and U.S. 3,835,113, and comprises extruding viscose which is a solution of sodium cellulose xanthate in caustic soda through an annular die into a coagulating bath to form a tubular casing.

The viscose solution is prepared by steeping chemically pure cellulose, typically of a wood or cotton source in a concentrated caustic soda solution from which an alkali cellulose crumb is obtained. The alkali cellulose crumb is converted to cellulose xanthate crumb by reaction with carbon disulfide. After the reaction, the cellulose xanthate crumb is slurried with an aqueous dilute caustic soda solution in a proportion to yield from about 7 to 8% cellulose and a total alkalinity of from about 6 to 7% to form the viscose. The viscose usually has a degree of polymerization (D.P.) in the range of about 450-750.

The tubular casing, once extruded, is coagulated substantially immediately by passing the extruded casing into an aqueous bath maintained at a temperature of about 42°C containing about 250 grams per liter (g/l) sodium sulfate and from about 100-135 g/l sulfuric acid.



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The salt/acid mixture effects decomposition of the xanthate and coagulation of the extruded product. Coagulation liquid is applied both to the outer and interior wall surfaces to effect regeneration of the viscose. The casing is washed and then is plasticized by passing it through a water bath containing a plasticizer such as glycerine or a food acceptable glycol such as propylene glycol or diglycerol. The plasticized gel film is inflated under substantial air pressure and passed through a dryer to remove a substantial portion of the water to produce the finished casing product.

The film wall of casings used for the processing of sausages normally ranges in thickness from about 1.0 to 1.4 mil. The amount of cellulose material per unit length of the dried casing is conveniently indexed in the sausage casing art in terms of the weight of cellulose expressed in grams per 10 meters length (g/10m) of a given casing width range or "Bone Dry Gauge" (BDG). The BDG of a casing will vary with the diameter of the casing as may the thickness. On the basis of commercial performance tests, the BDG for a casing having a dry flat width of 22.0-23.0 millimeter (mm) (Code 25) used for the processing of frankfurters is normally 20 g/10m. The flat width of dried regenerated cellulose casings normally ranges from about Code 13 (21.0 mm) to about Code 40 (dry flat width 51.3 mm). dry flat width range the basis weight of the casing normally ranges from 24 to 34 grams per square meter (q/m^2) and the BDG ranges from 10.2 to 31.2 g/10m.

The morphology or macrostructure of the regenerated cellulose casing wall is composed primarily of an outer skin portion on both the exterior and interior sides of the wall, the skin being characterized by large, poorly ordered amorphous regions and small or imperfect crystalline regions and an inner core portion characterized by large crystalline regions separated by amorphous regions of fairly high order, the skin and core sections of the wall structure being readily distinguished from each other when samples of a torn dehydrated section are examined under a scanning electron microscope (SEM), the skin portion being tougher than the core.

It has been determined that the wall of a cellulose



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casing having a BDG of 20 g/10m made under conventional viscose process conditions will have a skin layer forming about 7 to 25% of the total casing wall cross-sectional area.

After the coagulated and regenerated gel casing is impregnated with a plasticizer, it is dried to form the final casing product. Thereafter, the casing is wound on reels and subsequently shirred on high speed shirring machines, such as those described in U.S. Patents 2,010,626, 2,583,654, 2,722,714, 2,722,715, 2,723,201 and 3,451,827. In the shirring process, lengths of from 40-200 feet of casing are compacted into pleated strands of a few inches, e.g., 4-30 inches at a rate of 10 to 15 feet per second (ft./sec.).

After a casing is shirred, it is packaged and shipped to a meat packing house where an individual shirred strand is placed on a stuffing horn and a meat emulsion extruded to fill the casing to its fully extended length. The stuffing of the casing usually takes place within a few seconds with the result that the casing is extended from a shirred length of 8-27 inches (in.) to an extended length of 40-160 feet (ft.) or more at a rate of 2-6 ft/sec.

The shirring process imposes several score pleats per foot of casing at extremely high rates of loading so that the casing must be flexible enough to withstand such pleating without damage to the casing wall which later shows up as pinholes during high speed stuffing operations. The rapid extension of the casing during stuffing requires that the casing be especially strong and resistant to tearing. If even minor holes develop in the casing, the casing may split or break during stuffing with the disadvantageous loss of meat product.

It is therefore critical to the commercial utility of regenerated cellulose casings that the casing be sufficiently flexible to be shirred without damage and the resultant shirred strand must be able to be readily deshirred under high speed stuffing operations without substantial breaking or pinholing. The shirred casing strand must be of sufficient strength to withstand normal handling required for providing end closures in the casing and placement in high speed stuffing machines.

Typical regenerated cellulose casing contains 65%



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cellulose, approximately 20% non-volatile plasticizer and the remainder being water. The plasticizer is incorporated in the casing product in order to make the casing flexible enough to be shirred at high speeds without damage and to be stuffed at high speeds without pinholing breakage or other commercially unacceptable behavior. During the shirring operation the casing is humidified so that the final moisture content is 16 to 20%. moisture imparts additional plasticization to the casing and imparts greater flexibility to the casing to withstand subsequent high speed stuffing operations.

Economic incentive exists to substantially reduce the basis weight of the casing as well as to reduce or eliminate the plasticizer component. Efforts to reduce the basis weight of regenerated cellulose casing made under conventional viscose process conditions results in casing products which are of insufficient strength for shirring and stuffing under modern commercial conditions. Attempts to manufacture casing of standard basis weight, but with elimination of the plasticizer, results in a shirred casing exhibiting a 50% tensile strength loss which encounters unacceptable high breakage during high speed commercial meat stuffing.

Summary of the Invention

In accordance with the present invention a lower cost regenerated cellulose casing having improved casing physical properties is obtained with a regenerated cellulose casing of substantially reduced basis weight, e.g. 20-45% or lower, and a substantially reduced plasticizer content, e.g., 10% or less, the casing having the requisite strength and flexibility to undergo commercial shirring and meat stuffing operations without substantial breakage and pinholing.

The high strength, low plasticizer content, thin-walled shirrable regenerated cellulose sausage casing of the present invention is produced by modifying the known viscose process wherein the conditions under which coagulation of the extruded tubular casing are altered so that a reduction in the regeneration rate of the cellulose xanthate tubular extrudate relative to the coagulation rate is effected. Such modification generally comprises the use of a lower coagulation bath temperature,



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namely, in the range of 22° to 38°C, preferably 28° to 34°C, a decreased acid content in the order of 90 to 130, preferably 100 to 130 g/l, and an increased dehydrating salt content, namely, in the order of 200 to 300 g/l and preferably 250 to 280 g/l in the coagulation bath.

By the practice of the present invention, regenerated cellulose sausage casing having the flexibility and strength to withstand commercial shirring and meat stuffing operations is obtained having a reduced wall thickness of about 0.44 to about 0.87 mil as compared to prior art casings of 1.0 to 1.4 mil thickness and a reduced plasticizer content of 0-10% by weight, as compared to prior art casing plasticizer content of 18-25% by weight. The casings of the present invention have a substantially lower basis weight. For example, Code 25 casing of the present invention has a BDG of 12 to 15 g/10m as compared with a standard BDG of 20 g/10m in Code 25 casing.

The casing products of the present invention can be shirred and stuffed without appreciable increase in the defect level, e.g., in pinholing or strand breakage, being noted. The casing product of the present invention is appreciably denser than prior art casing due to the absence of plasticizer and the appreciable absence of voids normally present in prior art casing. The voids are caused by the generation of gaseous by-products during the coagulation and regeneration steps of casing manufacture as the relatively thick walls of the gel casing impede the rapid diffusive escape of such gases during normal casing processing.

By the practice of the present invention the skin layer of the casing wall may be increased to levels in the order of 30-50% of the total casing wall, the skin layer levels increasing as the temperature of the coagulation bath is decreased at a constant bath concentration. An increase in skin content effects a decrease in permeability of the casing to low molecular weight specie.

Preferred Embodiments

As previously described, the regenerated cellulose sausage casing of the present invention is produced by extruding a thin-walled extrudate of an alkaline viscose solution of



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conventional composition into a low temperature coagulation bath having a relatively low acid content and a relatively high dehydrating salt content. The alkaline viscose solution is prepared by conventional procedures well known in the art and advantageously contains from about 6 to about 8% by weight cellulose and about 5 to about 8% by weight total alkalinity, the cellulose xanthate being derived from an alkali cellulose crumb having a DP in the order of 450 to 750 and preferably 550 to 650.

The viscose solution once prepared is extruded through an annular die of the type conventionally employed for the extrusion of tubular casings at the rate of 100 to 180 feet per minute (fpm) and preferably 110 to 150 fpm into a Muller coagulation bath maintained at a temperature of 22° to 38°C, preferably 28° to 34°C containing from about 200 to 300 g/l and preferably about 250 to 280 g/l sodium sulfate and 90 to 130 g/l, preferably 100 to 130 g/l sulfuric acid. For a given contact time in the coagulation bath, the percent skin morphology increases as the temperature of the bath is decreased, as the salt concentration is increased, as the acid concentration decreased and as the xanthate content of the viscose is increased.

To achieve a reduction in casing wall thickness to prepare the thinner walled casing product of the present invention, the original width of the viscose process die through which the viscose is extruded is narrowed compared to the die orifice width used for conventional, thicker wall casing, i.e., the orifice width is reduced from about 11.5-12.0 mils to about 9 mils. Alternatively, and preferably, the reduction in casing wall thickness can be achieved simply by using a conventional extrusion die having 11.5-12.0 mils annular clearance and pumping the viscose to the die at a proportionately lower, e.g., 25 to 40% lower, volume rate, so that at a fixed machine speed, the extruded casing is drawn from the die at the same linear speed as the thicker casing but with less volume of material per unit length.

After extrusion, the coagulated tubing is withdrawn from the coagulation bath and passed into a water wash bath to remove carryover salts from the coagulation bath. The seamless



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cellulosic gel tubing thus formed is then successively passed through a series of conventional regeneration baths containing aqueous solutions of low concentrations of coagulation salts and acid and thereafter through a series of counter-current water baths to remove the acid and salts remaining in the casing. washed cellulosic tubing, which is still in a gel-like state can, if desired, be passed through a plasticizer bath composed of a solution of a water soluble hydroscopic plasticizer such as glycerine, sorbitol or a glycol such as propylene glycol. passage of the casing through the plasticizer bath is adjusted so that no more than about 10% by weight of the plasticizer is added to the gel casing. For many sausage casing applications, the plasticizer bath is eliminated entirely. The concentration of the plasticizer when present in the regenerated cellulose casing product of the present invention is generally less than about 10% and ranges from 0 to 10% by weight of the casing.

The washed gel tubing is then dried to obtain the final casing product. Drying is accomplished by passing the casing through a hot-air dryer of the type conventionally used in the viscose process which is comprised of a heating tunnel having paired sets of rotating squeeze rolls at the entrance and exit stations. Heated air is circulated in the tunnel and the gel casing is continuously advanced through the tunnel by means of the paired rotating entrance and exit rollers. An inflating quantity of air is confined within that part of the casing extending between the entrance and exit rolls. The exit rolls are driven at the same or slightly higher peripheral speed than the inlet rolls to effect longitudinal orientation. The casing is inflated to effect distention of the circumferential diameter, typically in the range of 12% to 60%. The temperature at which drying is accomplished is conventionally about 225° to 250°F. However, because of the reduced thickness of the casings of the present invention, drying, using the same conventional hot air driers, can be accomplished at temperatures 30° to 80°F lower, i.e., at about 150 to 200°F. The gel tubing is dried to a moisture content of about 6% to about 12% by weight and preferably 9% to 11% by weight.



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After drying, the cellulose casing is flattened in its passage through the exit rolls and is then wound on reels. The dried flattened casings then undergo conventional shirring operations by methods known to the art as previously discussed above, and the casings in the shirred state are shipped to the meat packer for the processing of sausage meats.

The regenerated cellulose sausage casing produced in this manner is a low basis weight, thin-walled (0.44-0.87 mil) casing having a low (0-10% by weight) plasticizer content, which in spite of the low plasticizer content is very flexible and can be easily handled during further processing, i.e., shirring, filling with sausage meat and tying off of the finished sausages.

While the low basis weight casing of the present invention, absent plasticizer content can be shirred without damage and stuffed at a commercially acceptable low defect level, plasticizer at a concentration of about 3 to 10% by weight is optionally incorporated in the casing to provide humectant or hydroscopiscity properties to the casing to retard the loss of moisture from the shirred strand when the strands are exposed to low humidity conditions. However, if adequate packaging and adequate control of the use of the casings in the packing plant is provided, the plasticizer addition is not required.

Because of the thinner wall dimensions of the casing, the casing can be shirred to produce a shirred strand which is 10 to 40% shorter for a given casing length than conventional shirred strands, resulting in a shirred product which is easier to handle and requires less packaging and storage space.

The low basis weight casing product of the present invention is stuffable with "frozen" i.e., 22° to 32°F meat emulsion at a very low defect level. Frozen meat emulsion is used for stuffing sausage products derived from turkey or chicken. The use of the frozen emulsion is necessary to minimize the incidence of microbiological activity during sausage processing. Conventional sausage casings formed from plasticized regenerated cellulose encounter a high defect level, e.g., 20 to 40% when stuffed with cold meat emulsion.



The invention will be further illustrated by reference to the following specific Examples.

Example I

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In a series of runs to prepare Code 25 casing, a solution of viscose having a DP of 600, containing 7.7% by weight of cellulose and 6.5% by weight calculated sodium hydroxide and having a viscosity of 72 falling-ball seconds at 18°C was extruded under pressure through an annular slot die at a rate of 110 fpm, a process during which it formed a tubular body. To achieve a reduction in basis weight, the viscose was pumped to the die lip at volume rates which were 25 to 40% lower than conventionally employed, e.g., 522 to 653 grams per minute (g/min) as compared to 870 g/min conventionally used to prepare Code 25 casing.

The tubing was extruded in different runs into a coagulation bath maintained at temperatures varying from 30.2°-36.0°C, the bath being composed of an aqueous solution containing from about 100-130 g/l sulfuric acid and from 255-272 g/l sodium The coagulated gel tube was withdrawn from the coagulation bath at the rate of 110 fpm and passed through a water bath at 25°C to remove occluded coagulation bath salts from the casing and then through several successive regenerating baths, the regenerating baths having a concentration of sulfuric acid and sodium sulfate which increased from 35 g/l by weight sulfuric acid and 100 g/l sodium sulfate in the first of these baths to 75 g/l sulfuric acid and 120 g/l sodium sulfate in the last of these baths. The gel casing was withdrawn from the terminal regeneration bath and passed through a series of counter-current flow water wash baths at a temperature of 35°-60°C. The washed casing was passed through a bath containing an 11% by weight glycerine water solution which had been adjusted to a pH of 7.0 with sulfuric acid. The time the casing was immersed in the bath was adjusted so that the final casing product would contain 0-20% by weight glycerine.

The plasticized tube was dried by passing the tube, in an inflated state through a hot air dryer, the air temperature being varied from 150°-260°F at the dryer entrance and maintained at 130°F and 118°F at the midpoint and end stations. After



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exiting the dryer, the dried casing was wound on a reel.

The dried casing product was Code 25 (22.0-23.0 mm diameter), had a moisture content of about 9% and was quite flexible. Measurements taken of the casings indicated that they had a BDG range of 12.2-15 g/10m. Examination under the SEM of torn dehydrated wall portions of the casing product indicated that the skin area of the casing wall ranged from 32.3-40.8% of the total cross-sectional area of the casing.

The process conditions used to prepare these casings are summarized in Table I below. The physical properties of the casing are summarized in Tables II-III.

In Table II the tensile and other physical properties were measured on one set of conditioned casing using an Instron tester. Conditioned casing was obtained by maintaining a casing in a room at 75°F and a relative humidity of 60% until moisture equilibrium with the atmosphere was attained. Conditioned casing physical properties give an approximation of casing characteristics under controlled moisture content.

In Table III the tensile and other physical properties of rewet casing is recorded. Rewet casing is casing wet in water and the tensile properties give an approximation of the casing characteristics during stuffing, after wet-out by the encased meat product.

For purposes of contrast, the procedure of Example I was repeated with the exception that either the coagulation bath temperature was 42.0°C (the temperature at which the viscose process coagulation bath is conventionally maintained) and the casing plasticized with 20% gylcerine (Run No. X) or the coagulation bath temperature was 42.0°C and the casing was not plasticized with glycerine (Run No. E) or the coagulation bath was lowered to 33°C but the casing was plasticized with 20% glycerine (Run No. K). The physical properties of these comparative casings are also summarized in Tables I-III below.



TABLE I Process Conditions

		Coa	agulati	lon	Temp.					
		Cor	ndition	ıs	(°F) at			Propertie		
	Run	Acid	Salt	Temp	Dryer	%	BDG	Basis, Wt.	. %	%
5	No.	g/l	g/l	°C	Entrance	H ₂ 0	g/10m	g/m ²	Skin	Glycerine
	A	130.5	255	33	180	9.3	15.0	21.8	28.0	20.63
	В	130.5	255	33	180	9.6	15.0	21.8	21.5	11.76
	С	124.0	257	32 .	180	10.2	15.2	22.1	35.9	. 0
	D	125.0	255	30.2	150	7.2	12.2	17.7	32.3	0
7 0	E	124.0	255	42.0	260	10.7	20.4	29.2	15.9	0
10	F*	124.0	255	36.0	205	9.5	15.1	21.9	15.6	12.0
	G	121.0	271	33.7	170	8.4	14.7	21.4	33.7	12.8
	Н	121.0	271	33.7	170	-	14.7	-	33.7	0
	I	103.0	270	30.3	140	13.8	14.7	21.4	40.8	12.6
	7	103.0	270	30.3	140	-	14.7	-	40.8	0
•	$K_{(1)}$	119.0	272	31.2	135	10.5	13.9	21.9	24.9	24.0
	$\Gamma(1)$	117.0	272	31.2	-	-	14.1	22.2	20.8	12.0
15	K(1) L(1) M(1)	114.0	171	29.6	_	-	13.7	21.6	26.0	0
	N	108.0	269	33.0	-	-	15.2	22.1	29.6	13.8
	0	102.0	269	33.4	-	**	14.8	21.5	32.2	0
	P	101.0	270	36.4	210	12.9	15.1	22.0	28-32	13.6
	X	127.0	147	42.0	210-220	9-11	20.3	29.2	14.0	20.0

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TABLE II
Conditioned (60%) Casing Tensile Properties (Flat Stock)

25	Break Tensile Run p <u>si</u>			Elongation %		Energy In.Lbs.	
	No.	Long.	Trans.	Long.	Trans.	Long.	Trans.
30	A B	18203 · 19847	15962 20227	27.7 19.9	67.1 53.9	6.247 4.597	7.570 7.457
30	С	23293 23746	24505 24187	13.8 12.5	41.4 41.7	3.424 2.419	6.460 5.090
	D E F	23691 20005	24773 19617	14.4 31.0	39.0 49.5	4.956 4.818	8.157 6.593
	I J K	16500 18900 13807	13490 19720 13216	26.0 16.0 27.5	67.0 57.0	- - 5 202	-
35	L M	17930 21747	17302 21018	21.0 14.0	72.3 53.8 38.3	5.101 4.395 3.071	6.321 5.571 4.267
	N O	-	15795 19189	-	63.0 43.0	-	7.25 6.16
	P X	- 18600	17671 13800	- 52.0	55.9 85.0	-	6.35

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^{20 * 125} ft/min extrusion speed, withdrawn from coagulation bath at same speed. * 145 ft/min extrusion speed, withdrawn from coagulation bath at same speed.

With reference to Tables I and II the tensile properties of conditioned casing made in accordance with the conventional viscose process (Run No. X) are such that the tensile strength in the transverse direction is substantially less (e.g., 60-90%) than that in the longitudinal direction.

In casings manufactured in accordance with the present invention the tensile strengths in both the longitudinal and transverse directions were substantially the same or isotropic. The isotropic character of the casing is desirable because when a casing is stuffed and linked with high speed stuffing machines, the links created in the casing by the twisting action used in linking imposes a circumferential stress on the casing. A high tensile strength in the transverse direction enables the casing to better withstand such linking stresses.

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TABLE III
Rewet Casing Tensile Properties

	Break Tensile		Elongation		Break Energy		
20	Run No.	Long. (psi)	Trans. (psi)	Long.	Trans.	Long. (In-Lbs.)	Trans. (In. Lbs.)
	A	4051	4885	44	46		
	В	4751	5776	46	106	3.64	- - 70
	C	5333	5561	46	97	3.91	5.18 4.32
	D	5010	5998	42	103	2.76	3.89
	E	5286	6174	50	99	5.61	7.17
25	F	4825	5791	47	110	4.18	5.79
23	H	5847	6299	47	105	4.54	5.50
	Ĵ	5155	5457	42	107	4.13	5.22
	K	3711	3779	34	85	2.64	2.66
	L	3802	4262	34	.86	2.22	2.86
	M	4156	4611	31	81	1.89	2.43
	N	-	5912	-	104	-	5.204
/	0	_	4598	-	87	-	4.03
30 (P	-	5543	•	88	. -	4.842
•	X	4500	4000	55	145	-	•

Energy is a measure of the toughness of the casing under the test conditions and is the area under Tensile-Elongation curve.



The data in Table III shows that the rewet tensile properties of low basis weight casing made in accordance with the present invention is equal to or greater than cellulose casing made in accordance with the conventional viscose process.

EXAMPLE II

The casings prepared in Example I were also subjected to the following evaluation tests: permeability, apparent porosity and density. The results of these tests are summarized in Table IV.

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TABLE IV

	Run No.	cm/hr	Apparent Porosity 2 /secx10-7)	Density g/cc
15				
	A	0.227	3.08	1.4652
	В	0.218	2.63	1.5222
	С	0.220	2.40	1.4904
	D	0.258	2.08	1.5014
	E	0.220	3.25	1.5102
		0.265	3.32	1.4712
20	F I	0.387	4.63	-
	J	0.328	3.59	-
	K	0.226	3.12	-
	L	0.260	3.21	-
•	M	0.239	2.83	•
•	N	0.399	5.01	-
	0	0.328	3.67	-
25	P	0.374	5.11	-
	X	0.175-0.214	3.15-3.85	1.342-1.359

Permeability is determined by the diffusion rate (centimeters per hour) of a 1% solution of potassium ferricyanide and was used to index the porosity of the casing wall to low molecular weight compounds.

Permeability is as measure of moisture evaporation from the encased frankfurter during processing as well as access to smoke to the meat during cooking and smoking of the encased meat product.

Permeability is the absolute measurement of the rate of permeation through the casing wall with no correction for wall thickness. Apparant porosity is a measure of the relative



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inherent porosity of the casing wall. Apparant porosity is obtained by multiplying the measured permeability by the wet thickness of the casing wall.

Density is determined by immersion of a casing section in xylene in a pycnometer. The higher the density the less microscopic or submicroscopic voids present in the casing. Higher densities also reflect the extent of highly ordered volume elements in the cellulose.

The data in Table IV indicates that although the low basis weight casings of the present invention were denser and had a measured permeability less than that of conventional , casing (Run No. X), the apparant porosity was equivalent due to the thinner walls of the low basis weight casing, wherein the low basis weight casings remain amenable to smoke and other gaseous casing agents used in sausage meat manufacture.

EXAMPLE III

The casings prepared in Example I were also subjected to the following additional evaluation tests: Rewet Flat width (RWFW), Recommended Stuffing Diameter Pressure (RSD), Burst Pressure and Burst Diameter. The results are summarized in Table V below.

TABLE V

	Run No.	RWFW (mm)	RSD Press (cm Hg)	Burst Pressure (cm Hg)	Burst Diameter (mm Hg)
25			···		
	A	31.9	10.9	24.9	36.3
	В	32.3	10.9	27.0	34.5
	С	33.6	7.8	26.7	33.2
. `	D	33.5	6.4	20.9	35.5
	E	33.4	9.6	35.8	33.9
	F	-	-	26.7	35.0
30	H	-	-	29.0	-
	J	-	•	29.3	_
	K	26.6	15.0	29.7	-
	L	27.5	14.2	31.2	_
	M	28.6	11.5	31.1	_
	N	32-33.5	19.8	27.3	-
	0	32-33.5	-	_	_
35	P	32-33.5	21.3	28.5	_
J J	X	38.2	12.8	32.5	-



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RWFW is the rewet flat width and is the width in millimeters that the casing acquires when wet in water for 20 minutes at room temperature.

The RWFW data in Table V indicates that the RWFW of casing prepared in accordance with the practice of the present invention can be controlled to present commercial standards.

Recommended stuffing diameter (RSD) is the diameter which the meat packer should stuff the casing to secure firm links of encased sausage meat having the required reproducible weight and ease of peeling of the casing from the sausage meat. Overstuffing results in excessing casing breakage and poor peeling. The recommended stuffing diameter of Code 23 is 20.5 mm and 22.5 mm for Code 25. RSD pressure is the pressure which the casing will be under when stuffed to the recommended stuffing diameter. It will differ with each code.

Burst pressure is the pressure to which the casing can undergo before burst. Burst diameter is the average diameter of the casing at burst. The burst diameter will be substantially larger than the recommended stuffing diameter and provides a leeway for pressure variations in the stuffing machine.

Burst pressure and burst diameter is determined by clamping the ends of a length of casing and introducing air rapidly into the casing. The expanded diameter is measured by a non-contact means and pressure is measured corresponding to each of the diameter measurements. The pressure at which the casing bursts is recorded in centimeters of mercury and the diameter at burst reported in millimeters mercury.

The data in Table V shows that in casing made in accordance with the practice of the present invention the burst diameter is hearly constant over a wide range of burst pressure. EXAMPLE IV

The Code 25 casings prepared in Example I were conditioned and then shirred using conventional shirring equipment. The tensile properties of the shirred casing were measured and these measurements are summarived in Table VI below.

The term "conditioned casing" means casing that has been exposed to constant humidity and temperature for a time sufficient for the casing to come to a moisture equilibrium with



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the humidified atmosphere. The casing when conditioned at 60% relative humidity (RH) will equilibrate after 24 hours at 72°F to a moisture content of about 12 to 14%, and at 80% RH at 72°F for 24 hours the casing will equilibrate to a moisture content of 18 to 20%.

The break tensile of unshirred conventional viscose process casing is normally in the range of 18,000-19,000 psi in the longitudinal (L) direction and 16,000-17,000 psi in the transverse (T) direction and an elongation of 45-50% in the longitudinal direction and 70% in the transverse direction. The unshirred conventional casing when subjected to rewet conditions has a break tensile of 5,192 psi in the longitudinal direction and 4,698 in the transverse direction and an elongation of 81% in the longitudinal direction and 146% in the transverse direction.

TABLE VI

Effect of Shirring on Tensile Properties

	Run		TENSILE	(psi)			ELONGATION	1 %	
	No.	60	% R _H	80%	RH	60%	RH	80%	RH
20		<u>L</u>	T	<u>r</u>	T	L	— _T	L	T
20	Ε×	=	=	9,674	11,626	=	=	$3\overline{4}.2$	32.8
	N	17,400	-	15,020	-	28.7	-	32.0	-
	0 .	22,140	-	19,340	-	16.2	-	22.0	-
	P	19,,264	-	16,660	-	20.0	-	29.0	-
	X	-	-	18,000-	16,000-	-	-	40.0-	65 . U
				19,000	17,000			45.0	
25 .				Under	Rewet Cond:	itions			
	С	-	-	- 6,013	6,905	_	-	47.0	101.0
	D	-	-	6,013	6,140	-	-	48.0	97.0
	M	•	-	5,921	6,000	-	-	44.0	86.0
	N	-	-	-	5,981	-	-	•	107.0
	X	-	-	4,935	4,740	-	-	50.0	98.0

As Manufactured Casing

* Break Tensile and Elongation in unshirred state were as follows:

Break	Tensile (psi)	Elongation	(%)
Ī	<u>T</u>	L	T
23,690	23,773	$1\overline{4}.4$	$3\overline{9}.0$

The data in Table VI indicates that the tensile properties of conventional casing (Run X) and low basis weight casing prepared in accordance with the practice of the present invention (Runs N, O, P) do not undergo substantial loss of tensile properties when shirred, whereas as glycerine-free cellulose casing



having a normal BDG level, e.g., 20.4 g/lom, (Run No. E) encounters a substantial, e.g., 50%, reduction in tensile strength after shirring.

The rewet data in Table VI indicates that adequate tensile strength is present for the thin-walled casing of the present invention even in the rewet state.

Example V

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Some casings prepared in Example I were shirred and subjected to meat stuffing operations.

In the manufacture of frankfurters, a meat emulsion is pumped into the shirred casing, the meat is cooked and cured in the casing and then peeled away from the cooked meat to obtain a "skinless" frankfurter. To enhance the peelability of the casing, a "peel coating" is sprayed onto the interior of the casing during the shirring operation.

During the shirring operation a peel composition composed of 0.75% carboxymethyl cellulose, 1.0% sorbitan trioleate and 0.05% polyoxyethylene (23) lauryl ether, the balance being water was sprayed onto the interior surface of a portion of the casings prepared in Example I following the procedure disclosed in U.S. 4,137,947. The casing was then shirred using the apparatus described in U.S. 3,451,827.

The resistance of the casings, both shirred and flat stock, to bursting under stuffing conditions was measured by the Mullen burst test.

Mullen burst tests were performed on dry, i.e., 10% moisture content casing and on conditioned casing having a moisture content that would be typical for a shirred casing to be stuffed. The Mullen bursts were conducted to determine whether the shirring operations would cause a reduction in the burst strength as compared to conventional casing. Typically, the casing of the present invention had a Mullen burst strength per mil thickness in the conditioned state that was higher than that for a plasticized conventional casing, thereby indicating that the reduction in basis weight in the casings of the present invention is offset by the increase in casing wall strength resulting from the reduction in plasticizer content and greater density. The data is summarized in Table VII below.



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TABLE VII

Casing Physical Properties

Mullen Bursts for Flat Stock and Shirred Strand

	Run. No.	% Moisture	psi	psi/mil thickness
5	· · · · · · · · · · · · · · · · · · ·			
	Α	9.9	52.0	68.1
	В	10.7	59.6	88.1
	Ba	15.1	45.0	67.2
	С	13.3	56.5	93.7
	Ca	27.5	50.4	82.6
	D	14.4	49.1	104.6
1.0	Da	24.9	42.5	90.4
10	E	12.2	80.6	98.3
	Еb	17.9	54.5	67.3
	F	9.4	57.5	83.0
	Fb	20.9	43.4	62.4
	G	16.3	43.3	72.6
	I	16.5	42.0	71.2
	K	17.7	42.1	50.1
15	M	16.6	49.1	46.7
	N	14-17	33.9	39.4
	0	14-17	42.1	59.3
	P	14-17	35.2	45.7
	X	14.0	50.9	46.2

"a" denotes that the casing is shirred to a 10% shorter strand, i.e., because casing wall is thinner casing can be compressed to a greater degree to produce a shorter strand.

"b" denotes that the casing is shirred using normal compression. All runs not denoted were measured using flat stock. EXAMPLE VI

Coated shirred strands prepared in Example V having a moisture content in the commercial range, e.g., 18-21%, were stuffed with sausage emulsion of various compositions used for making frankfurters. The shirred casing strand used was compressed to varying lengths from normal to 30% less than normal. The number of casing defects, i.e., primarily the existance of pinholes, was determined by observing the performance of the strands during shirring and stuffing operations. Shirring of the casing to compressed strands shorter than normal appeared to cause no damage to the casing.

Stuffing was accomplished with a Frank-A-Matic stuffing machine at 130-260 ft/min. using different meat emulsions. The



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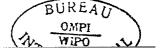
temperature of the meat emulsions was 57°F. The resulting stuffed links were of very uniform diameter. When the meat was cooked and smoked at a normal cycle in conventional smoke houses, a normal color, odor and taste resulted in the final cooked sausages. During the high speed stuffing operation, the casing showed normal pinhole tendencies and broke in excessive amounts only if stuffing was attempted at one to three millimeters above the recommended stuffing diameter. In normal meat packing operations, overstuffing to lmm above RSD is rarely encountered.

During the cooking process, which was a conventional cook cycle lasting about one and three quarter hours, the yield of weight of frankfurters from a given casing length did not differ materially from that of control of stuffed raw meat in conventional thick walled sausage casings.

The results of the stuffing tests are summarized in Table VIII below.

TABLE VIII
Meat Stuffing Test Results

	Heat bettiring rest he.	0 1 1 1/4 - +		
Run No.	Strand Length	Defects [*]	Cooked Meat Yield %	
Α	Normal	1	90.3-92.2**	
		0		
•			•	
D		1	89.2-92.5**	
	-10%	0		
	-20%	0		
	-30%	0		
F	Normal	1.	89.7-92.2**	
	-10%	0		
		0		
В		0	79.8-84.3***	
· ·		0		
•				
С			82.9-85.1***	
-				
E			86.6***	
2				
G				
A	-20%	U		
	A D	Run No. Strand Length A Normal -10% -20% D Normal -10% -20% -30% F Normal -10% -20% B Normal -10% -20% C Normal -10% -20% E Normal -10% -20% E Normal -10% -20% F -20% F -20% F -20% C -20% F -20%	Run No. Strand Length Defects A Normal 1 -10% 0 -20% 0 D Normal 1 -10% 0 -20% 0 -30% 0 F Normal 1 -10% 0 -20% 0 E Normal 0 -20% 0 E Normal 1 -10% 0 -20% 0 E Normal 1 G -20% 0 K -20% 0 K -20% 0 K -20% 0 N -20% 0 N -20% 0 P -20% 0 P -20% 0	



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breaks per 4 strands
beef-pork, 3% salt
100% pork, 2% salt
all pork, 3% salt

Control yield for beef-pork 3% salt is 88.1-91.2. Control yield for all pork was 86.6.

The data in Table VIII shows that the low basis weight. cellulose casing of the present invention having no plasticizer content can be stuffed at the normal 18 to 20% moisture content with an acceptable defect or breakage level and to produce links of standard cooked meat yield.

By way of contrast, Run E casing manufactured without plasticizer under normal (42°C) coagulation conditions showed a relatively higher defect level during commercial high speed stuffing operations.

15 EXAMPLE VII

The procedure of Example VI was repeated using a beef-pork emulsion with the exception that the temperature of the emulsion was lowered to 22-25°F to simulate frozen turkey stuffing. The results are recorded in Table IX below:

TABLE IX
Frozen Meat Stuffing Tests

	Run No.	No. of Strands	Defects
25	N	3	Break occurred at 26mm*
	O ኍኍ	2	0
	P	10	0***

* RSD is 22-23 mm

45°F meat emulsion

*** Frequent overstuffing occurred

EXAMPLE VIII

The procedure of Example I was repeated with the exception that the coagulation bath was maintained at 38°C to produce 16 g/10m BDG casing. The casings were plasticized with glycerine to levels ranging from 0-20% by weight. The skin content of the casings was 18-20%. The dried casings were shirred and humidified to a moisture level of between 13.5% to 20% water.

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The humidified shirred strands were stuffed at a meat packing house for making smoked sausages. Stuffing was accomplished at 130-260 ft/min.

The temperature of the beef-pork meat emulsion was 57°F. The color of the cooked, smoked casing was normal. Cooked yields were in the order of 90.6-92.1%. The number of casing defects, i.e., the existance of pinholes, was determined by observing the performance of the strands during shirring and stuffing operations. The results of the stuffing tests are recorded in Table X below.

For purposes of contrast, the procedure of Example I was repeated with the exception that the coagulation bath temperature was raised to 42.5°C, the temperature at which the coagulation bath is conventionally maintained. The casing had a BDG of 20.3 g/10m and the skin content was 14%. The color of the smoked, stuffed casing was normal. The number of defects is also recorded in Table X below. The comparative run is designated by the symbol "C" in Table X.

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TABLE X

Run. No.	Glycerol	H ₂ O	Strands	Defects
	Content %	Content %	Tested	Noted
1 2 3 4 5 6 C	20 20 10 0 0	20 14 20 20 17.5 13.5 16-20	2 1 2 4 1 1 2	0 0 0 0 0

EXAMPLE IX

The procedure of Example VI was repeated with the exception that the interior surface of the casing (Run Nos. 8, 9, Table XI below) was interiorly coated with the peel coating formulation used in Example V, during the shirring operation, or the interior of the casing (Run Nos. 10,11, Table XI below) was humidified with a formulation composed of a 1% water solution of sorbitan trioleate during the shirring operation. The casings were plasticized with different levels of glycerol. All the



casings had a moisture level of 16-18%. The number of defects found in the stuffed strands is recorded in Table XI below.

TABLE XI

5	Run No.	Glycerol Content %	Strands Tested	Defects Noted
	8	0	3	0
	9	10	3	0
	10	0	3	0
	11	10	3	0

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- 1. An artificial sausage casing of regenerated cellulose adapted to be shirred and stuffed with meat wherein the basis weight of cellulose of which the casing is comprised is in the range of about 16 to about 22 grams/square meter of cellulose for a casing dry flat width ranging about 21 millimeters to about 51 millimeters.
- 2. The casing of claim 1 wherein the casing has a plasticizer content of less than about 10% based on the weight of the casing.
- 3. The casing of claim 1 wherein the wall thickness of the casing is in the range of about 0.44 to about 0.87 mils.
- 4. The casing of claim 1 wherein the casing has a skin cross-sectional area wall structure in which the skin comprises at least about 30% of the cross-sectional area.
- 5. The casing of claim 3 wherein the skin comprises about 30 to about 55% of the cross-sectional area of the wall and has a plasticizer content of less than 30% based on the weight of the casing.
- 6. The casing of claim 1 wherein the casing is substantially free of plasticizer content.
- 7. In the method for producing tubular sausage casings of regenerated cellulose wherein viscose is annularly extruded to form a tubular product, the tubular product coagulated in a coagulation bath, the coagulated tubular product regenerated to form gel tubing and the gel tubing dried to form the casing product, the improvement comprising maintaining the coagulation bath at a temperature of about 25° to about 38°C.
- 8. The method of claim 7 wherein the gel tubing is plasticized to a plasticizer content between 0 and 10% by weight of the casing.
- 9. The method of claim 7 wherein the coagulation bath is comprised of about 200 to about 300 g/l sodium sulfate and 90 to 135 g/l sulfuric acid.
- 10. The method of claim 7 wherein the coagulation bath is comprised of 100 to 130 g/l sulfuric acid and about 250 to about 280 g/l sodium sulfate.



INTERNATIONAL SEARCH REPORT

International Application No PCT/US82/00142

	. •	International Application No PCT/US82/00142					
		N OF SUBJECT MATTER (if several class					
Accordin	ng to internat	ional Patent Classification (IPC) or to both Na	ational Classification and IPC				
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U.S.	J.S. Cl. 138/118.1, 264/196, 198; 493/272, 274, 293						
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Classificat	tion System		Classification Symbols				
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		493/272, 274, 293					
		Documentation Searched other to the Extent that such Document	than Minimum Documentation s are included in the Fields Searched 5				
III. DOC	UMENTS C	ONSIDERED TO BE RELEVANT 14					
ategory •	Citati	on of Document, 16 with indication, where app	propriate, of the relevant passages 17	Relevant to Claim No. 18			
X	US,A,	2,999,756, published SHINER ET AL.	12 September 1961,	1-10			
A .	US,A,	4,169,163, published JUDD ET AL.	25 September 1979,	1-10			
A	US,A,	3,818,947, published ROSE	25 June 1974,	1-10			
	-	f cited documents: 15					
		the general state of the art but published on or after the international	"P" document published prior to the li				
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IV. CERT	IFICATION		· <u>· · · · · · · · · · · · · · · · · · </u>				
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