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Formulation, optimization and texture characterization of environment friendly gum

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ABSTRACT

Chewing gum cause waste management problems around the world.Many consumers of chewing gum do not dispose product properly and gum is littered in public places.The syenthetic polymer based chewing gum have adhesive texture which help it to stick to various places and make it difficult to clean of surfaces .Their is constant need to modify chewing gum base in such a manner that they are chemically degradable. In current study corn zein,which is a major corn endosperm protein is used to make chewing gum.Various plastisizer are used for formulation of corn zein gum.Corn zein shows sufficient biodegradation property.In current study charectarization of chewing gum is performed using texture analyser and results are reported.

Key words: Corn zein, plastisizer, gumbase, chewing gum, biodegradable property

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INTRODUCTION

1. Formulation of Corn Zein chewing s gum

Zein is the water-insoluble prolamine from corn gluten, manufactured initially as a concentrated powder. It is unique in its ability to form odorless, tasteless, clear, hard and almost invisible edible films. Since zein films are completely safe to ingest, it is the perfect coating for foods[1-3] and pharmaceutical ingredients.

Zein is extracted from gluten by physical means and is, therefore, totally natural. It is a food ingredient, not an additive.Zein is shown to be remarkably resistant to bacterial attack, which frequently decomposes other proteinaceous material. It seems to repel many insects such as the Indian Meal Moth,which often infests nut and grain products that are unprotected.Zein is used commercially as adhesive,binder,biodegradable plastic,cosmetic powder etc. [4-10]

The quality of zein that makes it a prolamine, i.e., it's insolubility in water, insolubility in anhydrous alcohol, and solubility in a mixture of the two, is considered due to the preponderance of hydrophobic acids; leucine, proline and alanine. Zein insolubility in water is also due to the high proportion of hydrocarbon group side chains, and the high percentage of amide groups present with a relatively low amount of free caroboxylic acid groups.Zein is also used as delivery system for acid sensitive drug.[11-12]

Another characteristic of zein is its resistance to hydrolyzing by very dilute acids. In whole corn, zein occurs as a heterogenous mixture of disulfide - linked aggregates. Commercial extraction results in a product with molecular

weights of 25-35,000. It was discovered quite early that if the aqueous alcoholic solutions of zein were evaporated, a clear, hard film was formed, which, among other things, was completely edible and had many remarkable qualities. These films could be laid down as coatings on food and pharmaceutical ingredients offering substantial protection, as well as many other benefits.

MATERIALS AND METHODS

2.1. Ingredients for preparation of gum sample

Different formulations of corn zein chewing gum sample were included in the study, which varied in the plasticizer used. The corn zein gum formulations used Triacetin, Oleic acid ,PEG-600,Tributyl citrate, PEG-200, PEG-300, PEG-400, PEG-1000, Triethyl citrate and Castor oil as plasticizer. Other than the plasticizer, all of the ingredients and the amounts of each ingredient were the same for each formulation.

The ingredients used in making each of the corn zein chewing gums consisted of corn zein (regular grade M P Biomedical,LLC), ethanol (C.D.H. New Delhi), Sodium dodecyl sulphate (Estelle Pvt Limited), partially hydrogenated palm oil (Krishna Oil extraction limited, pachor, Rajgarh, M.P.), artificial cinnamon flavor (GLEE Gum kit U.S.A.), sorbitol solution (C.D.H. New Delhi), and plasticizer, Triacetin, Oleic acid , PEG-600, Tributyl citrate, PEG-200, PEG-300 ,PEG-400, PEG-1000,Triethyl citrate and Castor oil from (C.D.H. New Delhi). Corn zein is a food-grade protein, and all the other ingredients used in the gum formulation for this study were also food-grade quality. Table 1 shows a summary of the corn zein gum formulation.

Table 1: Summary of the Corn Zein gum formulation.(MCG1-MCG-10)

S.No	Ingredient(%w/w)	MCG 1	MCG 2	MCG 3	MCG 4	MCG 5	MCG 6	MCG 7	MCG 8	MCG 9	MCG 10
1	Corn Zein	50	50	50	50	50	50	50	50	50	50
2	Sodiumdodecyl sulphate	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
3	Palm Oil Partially Hydrogenated	8	8	8	8	8	8	8	8	8	8
4	Plasticizer	A 70	E 70	F 70	G 70	H 70	D 70	B 70	C 70	I 70	J 70
5	Sorbitol Solution	50	50	50	50	50	50	50	50	50	50
6	Filler Photoactive Titanium dioxide	5	5	5	5	5	5	5	5	5	5

Corn zein gum was made either with (A) Triacetin, (B) Oleic acid and (C) PEG 600,(D) Tributyl citrate, (E) PEG 200,(F) PEG 300,(G) PEG 400,(H)PEG 1000,(I)Triethyl citrate and (J) Castor oil

The exact amounts of each ingredient for the sample steps are summarized in Table 1.

2.2. Method for making corn zein chewing gum samples

Laboratory sigma blade mixer with front to rear speed ratio of 2:1 was used for formulation of Corn Zein chewing gum.Aqueous ethanolic solution of zein powder was poured in sigma blade mixer ,all the ingredients except hydrogenated soybean oil and flavor were added and mixed in sigma blade mixer for ten minutes.Sigma blade mixer had a temperature control device which maintains temperature intermittently until it reached 50 degrees Celsius. The special (z) shape of blade present in sigma blender helped in complete mixing and produce heat which evaporated the ethanol present in the solution.To prevent exposure of heat partially hydrogenated vegetable oil was added to sigma blender.[13] The corn zein solution was poured into the container which had five liters of purified ice water having its temperature maintained at three degree Celsius.The cold water caused zein to precipitate from ethanol solution.A dough like consistency was formed and zein particles were able to aggregate together and entrap rest of the ingredients.The dough was kneaded and rinsed in containers of purified water for two times, 10 min each to form a flexible gum base. The kneading action of sigma blade blender further blended the ingredients and rinsed away any remaining ethanol. The gum base was then spread into a thin sheet with a roller and cut into strips of 5 g each. Each strip was approximately 4 cm long, 1 cm wide, and 2 mm thick. All the gum samples were stored at room temperature

2.3. Method for coating corn Zein formulations

Coating of MCG was done by liquid coating solution of sorbitol & glycerin. This mixture was heated at 60 degree Celsius for 15 min and allowed to mix uniformly. Gum pieces were dipped in the solution, and after a specified time interval of 1 min, (to allow the liquid to spread evenly over the piece), a dry powder material (Sorbitol) was applied. This helps to dry the liquid coating; this is referred to as Dry Charging & is commonly used in soft panning operation. This was applied in about 3 to 12 dry charge application. After a dry charge 2 to 4 liquid applications are made to cover dry charge material, then the coating was dried in the hot warm air in the temperature range 27 ^{0}C to 38 ^{0}C.Table 2 shows concentration of five coating solution.

Table 2: Concentration of coating solutions

S.no	Ingredients	Coating 1	Coating 2	Coating 3	Coating 4	Coating 5
1.	Gum base	25	25	25	25	25
2.	70% Sorbitol	15	6	6	51	51
3.	Glycerin	6	15	51	6	15
4.	Sorbitol	51	51	15	15	6
5.	Flavor	3	3	3	3	3

Observations-Coating hardness and interior hardness of corn zein formulation (MCG-1 to MCG-10) is summarized in (Table 3-5).Coating 1 and coating 2 solutions which comprises of sorbitol 51%(w/w),was reported with highest average coating hardness of 1.223kg for coating 1 and 0.864kg for coating 2.

Texture Analysis (TA) Settings

Sequence Title: Return to Start (Set Dist)
Test Mode: Compression
Pre-Test Speed: 1 mm/sec
Test Speed: 2.0 mm/sec
Post-Test Speed: 10.0 mm/sec
Target Mode: Distance
Force: 100gm
Distance: 9.5 mm
Trigger Type: Button
Trigger Force: 5.0 g
Stop Plot At: Start Position
Probe:HDB/BSW, set with bratzler
Points per second: 250

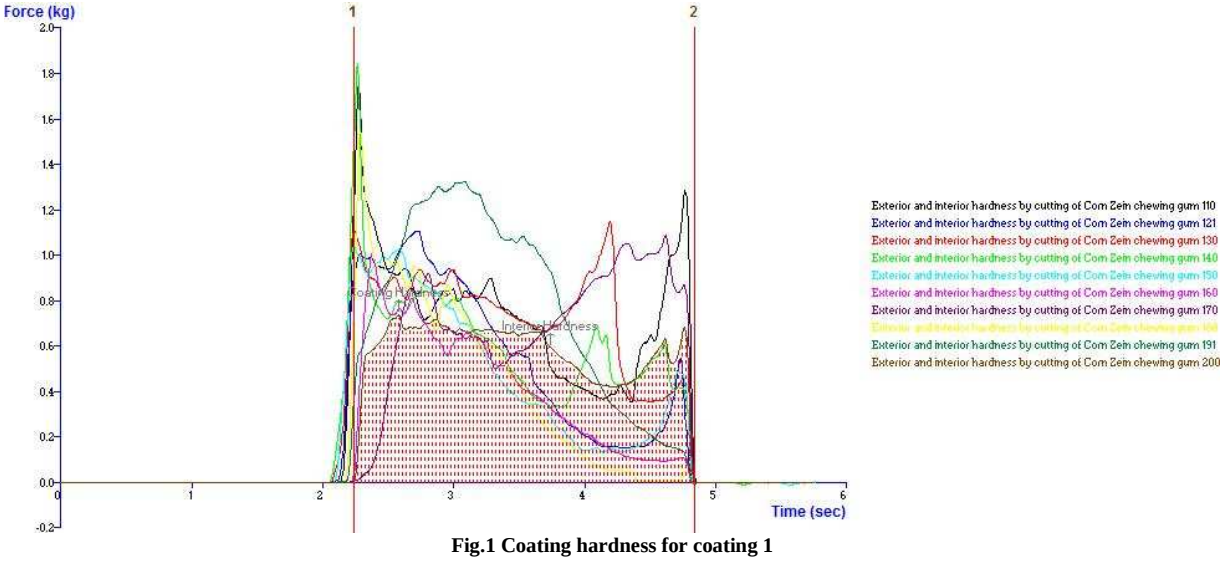

Fig.1 Coating hardness for coating 1

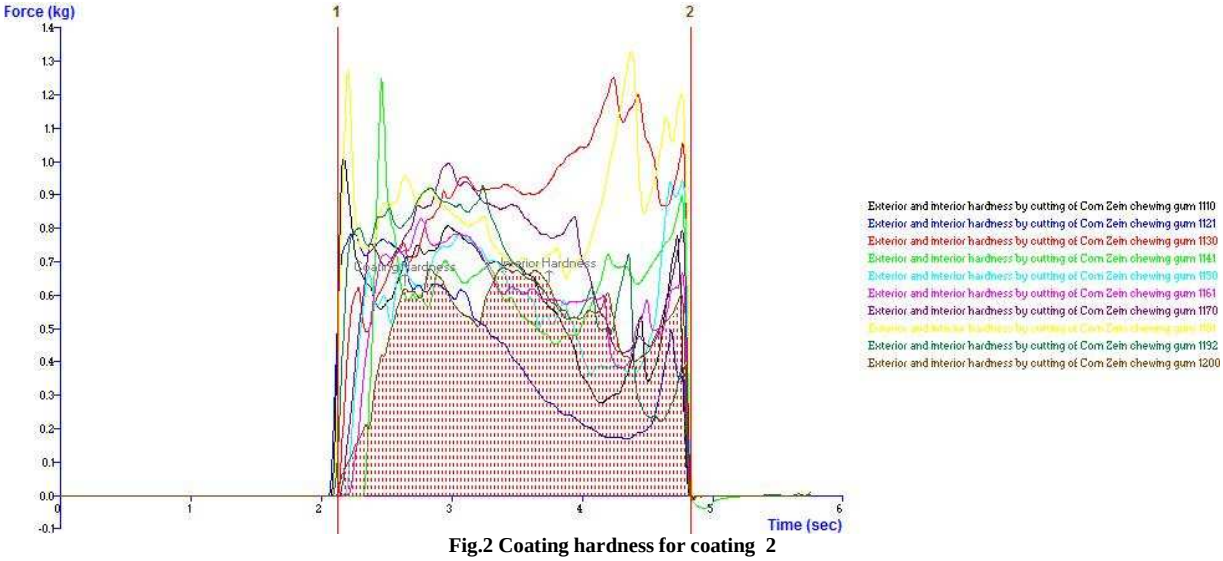

Fig.2 Coating hardness for coating 2

Coating 4 and coating 5 which comprises of sorbitol solution 51% (w/w),was reported with highest average coating hardness of 0.804kg for coating 4 and 0.757kg for coating 5.Decrease in coating hardness is due to use of high amounts of 70% sorbitol solution and 15%(w/w) glycerol solution in coating 5.Coating 3 shows average coating hardness of 0.864kg, and decrease in coating hardness value is due to use of high amounts of 51%(w/w) glycerin in coating 3.

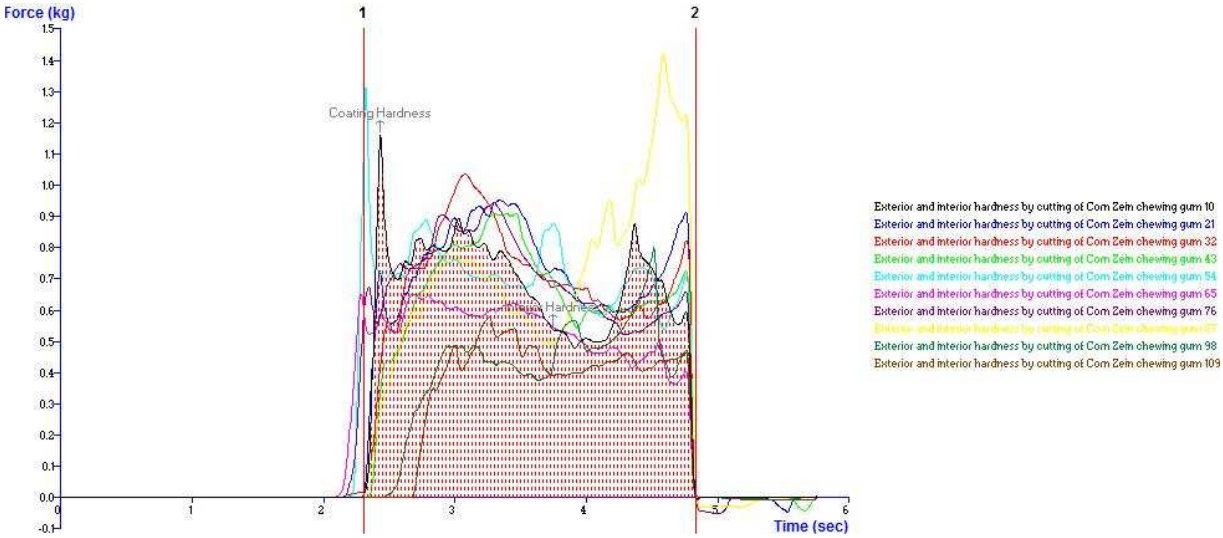

Fig 3. Coating hardness for coating 5

Table 3:Hardness obtained by coating 1.

Test ID	Batch		Coating Hardness Kilograms Kg	Interior Hardness Kilograms Kg	Area F-T 1:2,kg.sec
Start Batch 1	1				
Hardness by cutting of corn zein gum,MCG-1	1		1.755	0.501	1.985
Hardness by cutting of corn zein gum,MCG-2	1		1.008	0.357	1.526
Hardness by cutting of corn zein gum,MCG-3	1		1.109	0.680	1.984
Hardness by cutting of corn zein gum,MCG-4	1		1.848	0.336	1.686
Hardness by cutting of corn zein gum,MCG-5	1		1.038	0.281	1.373
Hardness by cutting of corn zein gum,MCG-6	1		1.017	0.324	1.093
Hardness by cutting of corn zein gu ,MCG-7	1		0.858	0.690	1.822
Hardness by cutting of corn zein gum,MCG-8	1		1.552	0.227	1.198
Hardness by cutting of corn zein gum,MCG-9	1		1.305	0.919	2.036
Hardness by cutting of cornzeingum,MCG-10	1		0.739	0.596	1.512
Average:	1(F)		1.223	0.491	1.622
S.D.	1(F)		0.379	0.224	0.337
Coef.of variation	1(F)	STDEV(“BATCH”)/AVERAGE (“BATCH”)*100	30.991	45.772	20.007

Table 4:Hardness obtained by coating 2.

Test ID	Batch		Coating Hardness Kilograms Kg	Interior Hardness Kilograms Kg	Area F-T 1:2,kg.sec
Start Batch 1	1				
Hardness by cutting of corn zein gum,MCG-1	1		1.016	0.568	1.610
Hardness by cutting of corn zein gum,MCG-2	1		0.623	0.632	1.394
Hardness by cutting of corn zein gum,MCG-3	1		0.786	0.281	1.225
Hardness by cutting of corn zein gum,MCG-4	1		0.624	0.942	2.392
Hardness by cutting of corn zein gum,MCG-5	1		1.253	0.479	1.579
Hardness by cutting of corn zein gum,MCG-6	1		0.668	0.526	1.546
Hardness by cutting of corn zein gu ,MCG-7	1		0.833	0.587	1.578
Hardness by cutting of corn zein gum,MCG-8	1		0.757	0.778	1.897
Hardness by cutting of corn zein gum,MCG-9	1		1.278	0.725	2.349
Hardness by cutting of cornzeingum,MCG-10	1		0.804	0.587	1.761
Average:	1(F)		0.864	0.611	1.733
S.D.	1(F)		0.241	0.179	0.382
Coef.of variation	1(F)	STDEV(“BATCH”)/AVERAGE (“BATCH”)*100	27.859	29.289	22.020

Table 4:Hardness obtained by coating 5.

Test ID	Batch		Coating Hardness Kilograms Kg	Interior Hardness Kilograms Kg	Area F-T 1:2,kg.sec
Start Batch 1	1				
Hardness by cutting of corn zein gum,MCG-1	1		1.159	0.539	1.696
Hardness by cutting of corn zein gum,MCG-2	1		0.727	0.731	1.834
Hardness by cutting of corn zein gum,MCG-3	1		0.563	0.673	1.752
Hardness by cutting of corn zein gum,MCG-4	1		0.911	0.698	1.632
Hardness by cutting of corn zein gum,MCG-5	1		1.311	0.878	1.839
Hardness by cutting of corn zein gum,MCG-6	1		0.651	0.547	1.397
Hardness by cutting of corn zein gu ,MCG-7	1		0.676	0.694	1.794
Hardness by cutting of corn zein gum,MCG-8	1		0.599	0.499	1.812
Hardness by cutting of corn zein gum,MCG-9	1		0.486	0.427	1.021
Hardness by cutting of cornzeingum,MCG-10	1		0.488	0.392	0.908
Average:	1(F)		0.757	0.608	1.568
S.D.	1(F)		0.283	0.152	0.345
Coef.of variation	1(F)	STDEV("BATCH")/AVERAGE ("BATCH")*100	37.347	24.970	22.013

2.4. Optimization- Optimization was done on the bases of texture profile analysis. Texture profile analysis (TPA) is an objective method of sensory analysis of compressing standard-sized samples of food twice. The test consists of compressing a bite-size piece of food two times in a reciprocating motion that imitates the action of the jaw and from the resulting force-time curve a number of textural parameters can be calculated, that correlate well with sensory evaluation.

The parameters derived from TPA test are as follows.

A) Hardness-Hardness is defined as the maximum peak force during the first compression cycle (first bite) and often been substituted by term firmness.

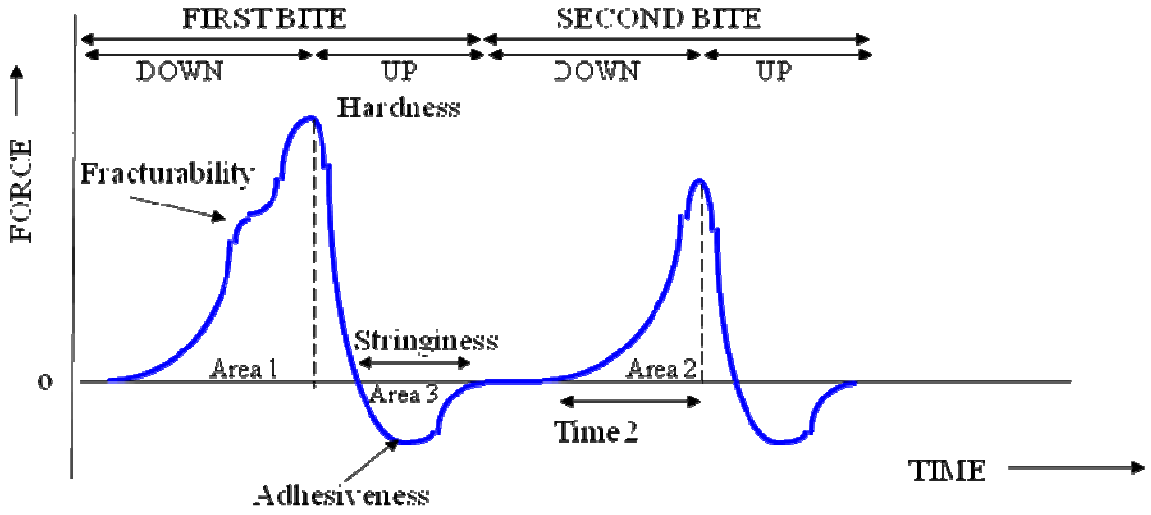

Fig 4: Texture profile analysis (TPA) graph showing various TPA parameters

Cohesiveness = area 2/area 1
Springiness = time2/time1
Gumminess = hardness x cohesiveness
Chewiness = gumminess x springiness

B) Fracturability (Originally called brittelness) is defined as the force at the first significant break in the TPA curve.
C) Adhesiveness –Adhesiveness is defined as negative force area for the first bite and represent the work required to overcome the attractive forces between the surface of the food and the surface of the other material with which the food comes into contact,i.e the total force necessary to pull the compression plunger away from the sample.

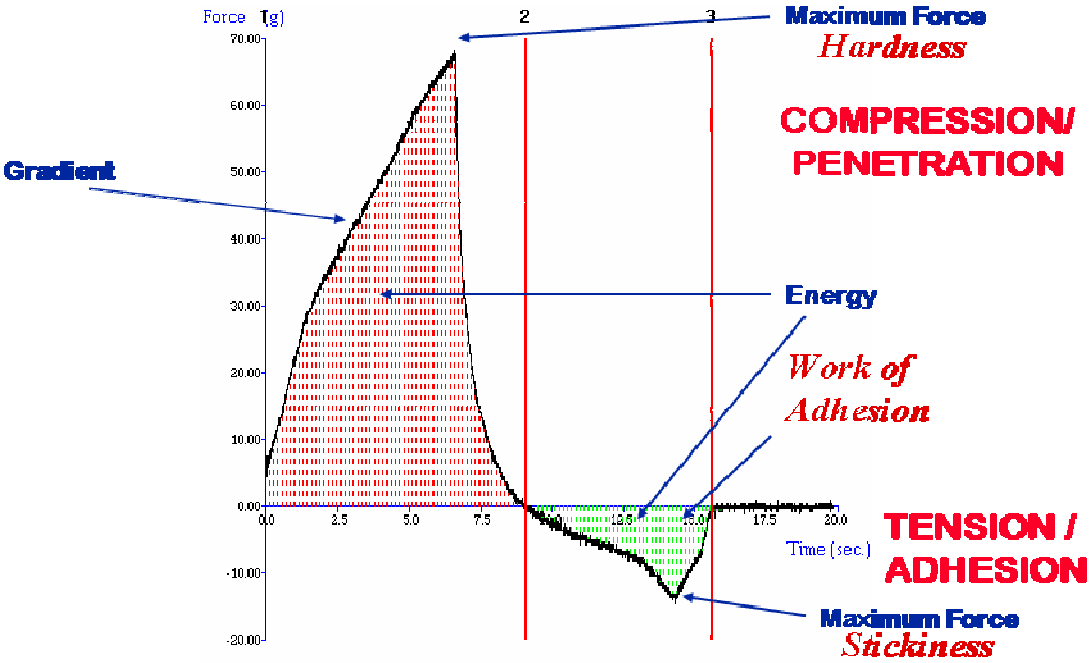

Fig 5: Texture profile analysis (TPA) graph showing various TPA parameters

D) Springiness (Elasticity)-It is related to the height that the food recovers during the time that elapses between the end of first bite and the start of the second bite.
E) Cohesiveness-It is defined as the ratio of the positive force area during the second compression to that during the first compression and may be measured at the rate at which material disintegrate under mechanical action.
F)Stringiness-It is the distance the product is extended during decompression before separating from compression probe.
G) Chewiness –It is measured in terms of the energy required to masticate a solid food and is calculated as the product of Hardness x Springiness x cohesiveness and should be calculated in TPA of solid food.
H) Gumminess-It is calculated as the product of Hardness x Cohesiveness and is characteristic of semisolid food ,with low degree of hardness and high degree of cohesiveness.

Different chewing gum samples were tested by using a Compression platen of 35 mm diameter (P/35) with Texture Analyser and Texture profile analysis (TPA test) was performed for measurement of properties like Hardness, Fracturability, Springiness, Cohesiveness, Adhesiveness, Gumminess, Chewiness and Resilience.

MCG-1,MCG-6,MCG- 7 and MCG-8 were selected as optimized batches as their values are similar to marketed preparations(Nicotine Polacrilex gum,Manufactured at Zenara Pharma private limited,Hyderabad and marketed by Johnson & Johnson Limited),they used Triacetin, Tributyl citrate ,Oleic acid and PEG 600 as plasticizer (Table 5) TPA Analysis of optimized batch MCG-1, which used Triacetin as plasticizers showed highest guminess,chewiness and cohesiveness values of 1133,326 and 0.224 respectively.These value obtained are similar to reference value

obtained from marketed formulations. TPA Analysis of optimized batch MCG-7, which used Oleic acid as plasticizer showed second highest guminess,chewiness and cohesiveness values of 705.7,199.43 and 0.185 respectively.Thus TPA analysis indicate that triacetin and oleic acid can be effectively used as plasticizer for corn zein formulations.(Table 6)

Table 5: Optimized Formulation of various corn zein chewing gum.

S.No	Ingredient	MCG-1	MCG-6	MCG-7	MCG-8
1.	Corn Zein	50g	50g	50g	50g
2.	Sodiumdodecyl sulphate	2.5g	2.5g	2.5g	2.5g
3.	Palm Oil Partially Hydrogenated	8g	8g	8g	8g
4.	Plasticizer	A 70g	D70g	B 70g	C 70g
5.	Sorbitol Solution	50g	50g	50g	50g
6.	Filler- Photoactive Titanium dioxide	5g	5g	5g	5g

Table 6:TPA values for various formulations

Test ID	Batch	Hardness gm force	Fracturability gm force	Adhesiveness gm.sec	Springiness Sec	Cohesiveness gm.sec	Guminess gm.sec	Chewiness gm.sec	Resilliance gm.sec
TPA,MCG-1	1	5056.711	2746.095		0.289	0.224	1133.039	327.063	0.115
TPA,MCG-2	1	6407.725			0.507	0.329	2106.712	1067.785	0.465
TPA,MCG-3	1	4617.168	2612.861	-0.032	0.587	0.244	1127.036	662.134	0.312
TPA,MCG-4	1	6846.028		-0.038	0.577	0.365	2500.852	1444.154	0.562
TPA,MCG-5	1	7019.737		-0.060	0.615	0.492	3453.286	2125..099	0.856
TPA,MCG-6	1	2664.95	2796.109		0.295	0.177	472.177	139.168	0.073
TPA,MCG-7	1	3806.81	2615.966	-0.233	0.283	0.185	705.706	199.439	0.093
TPA,MCG-8	1	2793.82	3130.644	-0.498	0.276	0.176	491.306	135.36	0.065
TPA,MCG-9	1	4862..826			0.410	0.256	1246.655	510.678	0.310
TPA,MCG-10	1	6891.005		-0.016	0.554	0.403	2776.137	1538.130	0.621
Coef.var		0.193	0.083	-0.774	0.159	0.274	0.437	0.544	0.410
S.D.		1144.362	228.646	0.047	0.085	0.092	913.805	638.362	0.205
Avg.		5936.004	2758.540	-0.061	0.536	0.338	2091.920	1173.257	0.500

2.5. Characterization of medicated chewing gum:

2.5.1. Physical evaluation of Medicated Chewing Gum: All Medicated Chewing Gum formulations were visually inspected; various physical properties of gum base were studied on basis of their solubility studies, relative humidity, color and moisture absorption. Following parameters were studied:

a) Physical evaluation of Corn Zein Gum: All formulation prepared by above procedure were physically evaluated for following parameters, Appearance, Color, Stickness,Hardness, and texture analysis.

b) Hardness/Resiiliance:. Texture analyzer was used for determining strength and degree of deformation. Values obtained indicate the flexibility of the sample.
c) Stickiness: Texture analyzer from stable micro system model TA.XT-EXPRESS was used for determining Texture profile analysis (T.P.A). Values obtained indicate uniformity of the sample.

2.5.2. Texture Profile Analysis (TPA) of Corn zein chewing gum [14]
Texture profile analysis (TPA) is an objective method of sensory analysis pioneered by Szczesniak.TPA is based on the recognition of texture as a multi parameter attribute.The test consists of compressing a bite size piece of food two times in a reciprocating motion,that intimates the action of the jaw and extracting from the resulting force time curve a number of textural parameters.

a) - Objective: Testing of chewing gum by cutting with HDB/BSW,Blade set with warner bratzler.
Texture Analysis (TA) Settings
Sequence Title: Return to Start (Set Dist)
Test Mode: Compression

Pre-Test Speed: 1 mm/sec
Test Speed: 2.0 mm/sec
Post-Test Speed: 10.0 mm/sec
Target Mode: Distance
Force: 100gm
Distance: 9.5 mm
Trigger Type: Button
Trigger Force: 5.0 g
Stop Plot At: Start Position
Probe:HDB/BSW,Blade set with warner bratzler
Points per second: 250

Test Set-Up:
Heavy Duty Platform was set onto the machine base. Sample was placed on the platform, centrally under the probe, and test was performed. It is important that regular shaped samples are selected and it is advantageous for the pellets to have a flat under side

Observations:
The probe approaches the sample and once the 5g trigger force is attained, a rapid rise in force is observed, as the probe cut/penetrates through the coating of the chewing gum. A drop in force is observed when the probe enters the interior of the gum. The probe returns to its original starting position when a penetration distance of 9.5mm from the trigger point is reached. The peak force is measured as an indication of the coating hardness. The force value at the distance of 9.5mm is considered as the interior hardness. Average coating hardness of 1.223kg for coating 1 (Table 7) and 0.877kg for coating 2 were obtained.TPA Analyis curve/GRAPHS for determining interior and exterior coating hardness of corn zein gum formulation (MCG1-MCG10) are illustrated below.(fig 6-fig16)

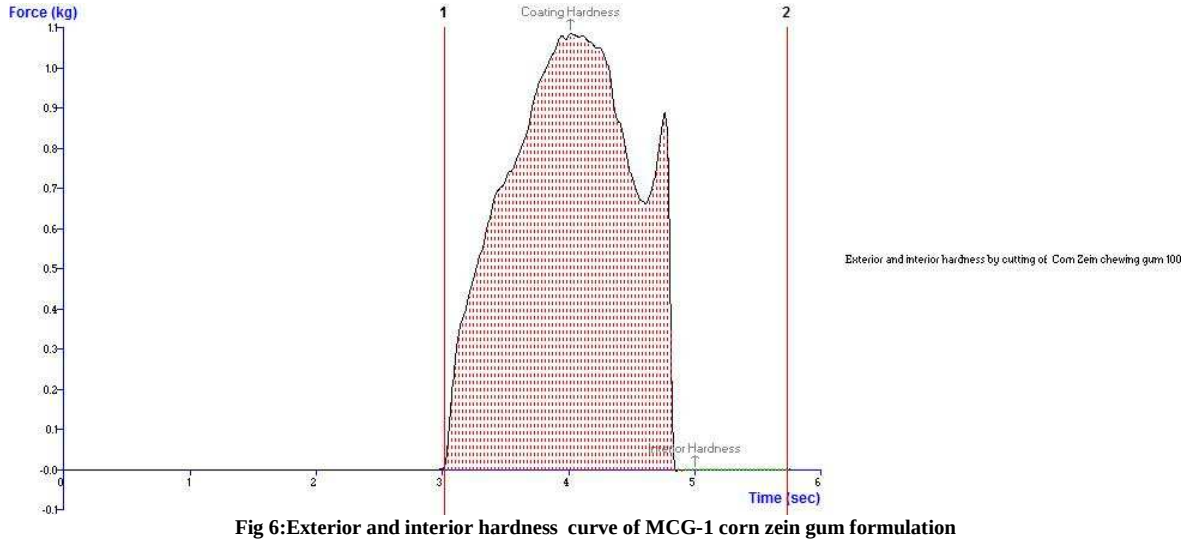

Fig 6:Exterior and interior hardness curve of MCG-1 corn zein gum formulation

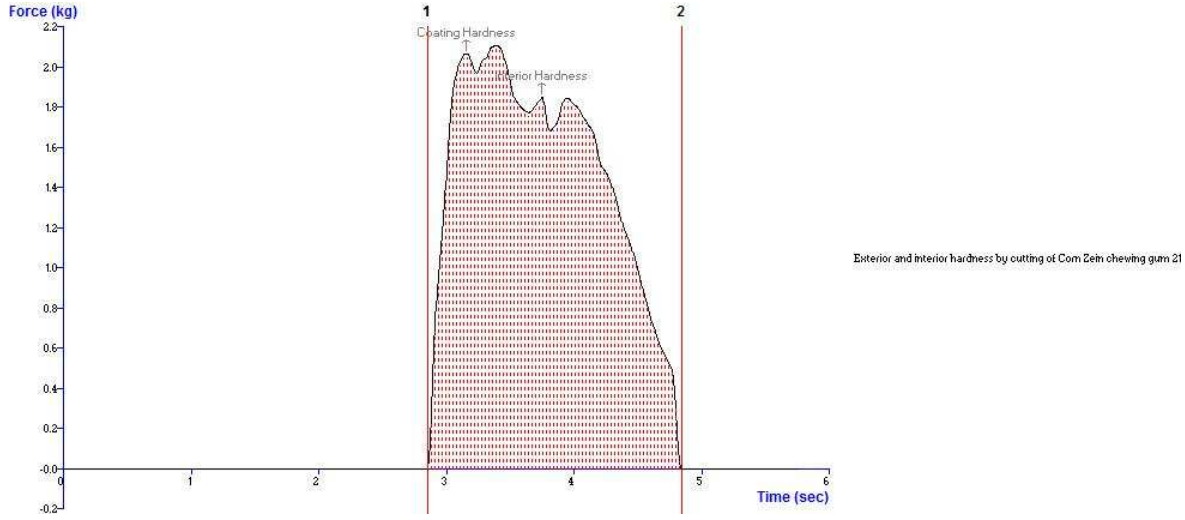

Fig 7: Exterior and interior hardness curve of MCG-2 corn zein gum formulation

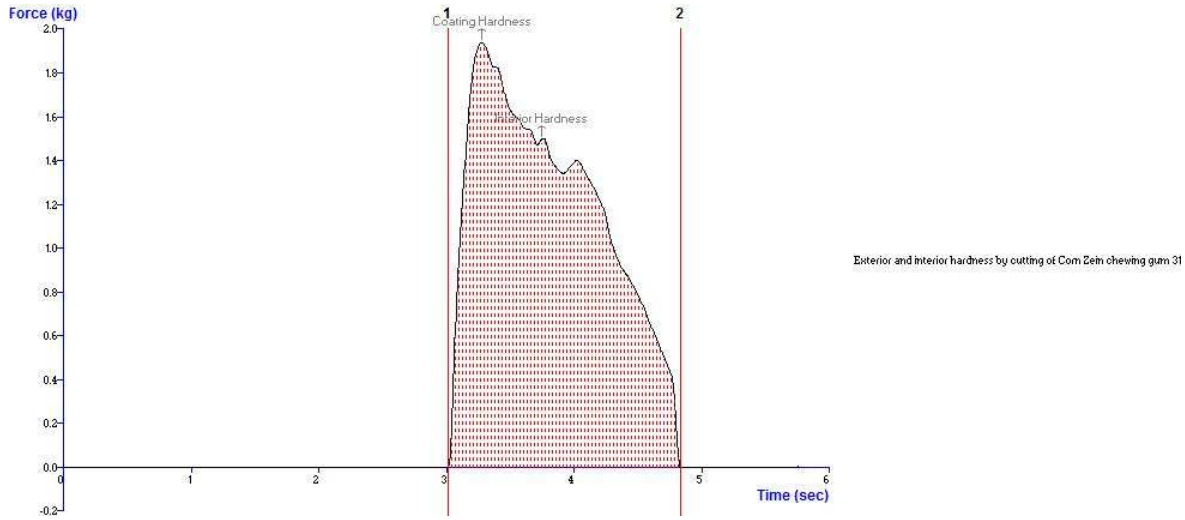

Fig 8: Exterior and interior hardness curve of MCG-3 corn zein gum formulation

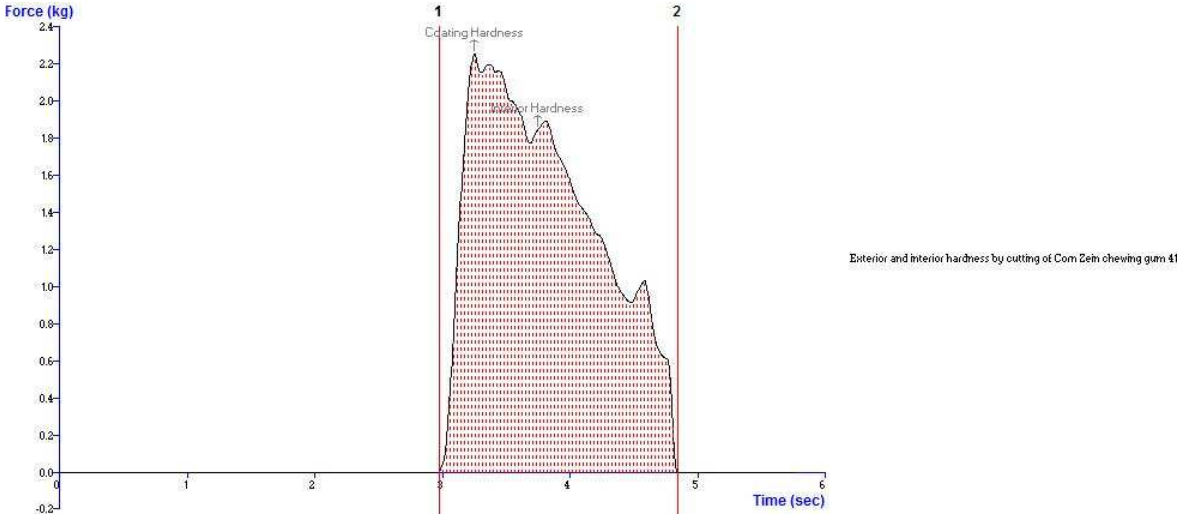

Fig 9: Exterior and interior hardness curve of MCG-4 corn zein gum formulation

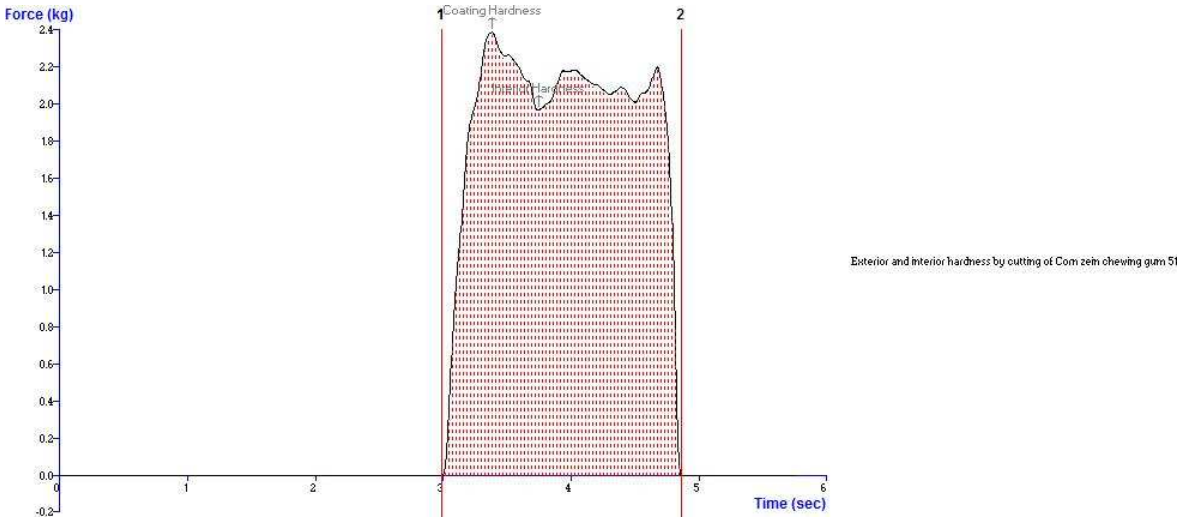

Fig 10: Exterior and interior hardness curve of MCG-5 corn zein gum formulation

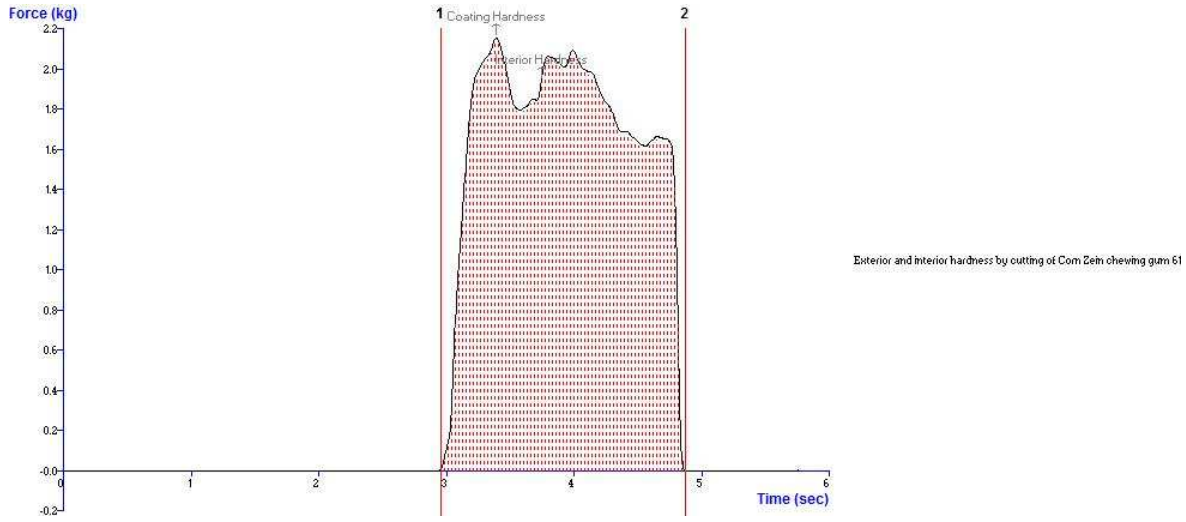

Fig 11:Exterior and interior hardness curve of MCG-6 corn zein gum formulation

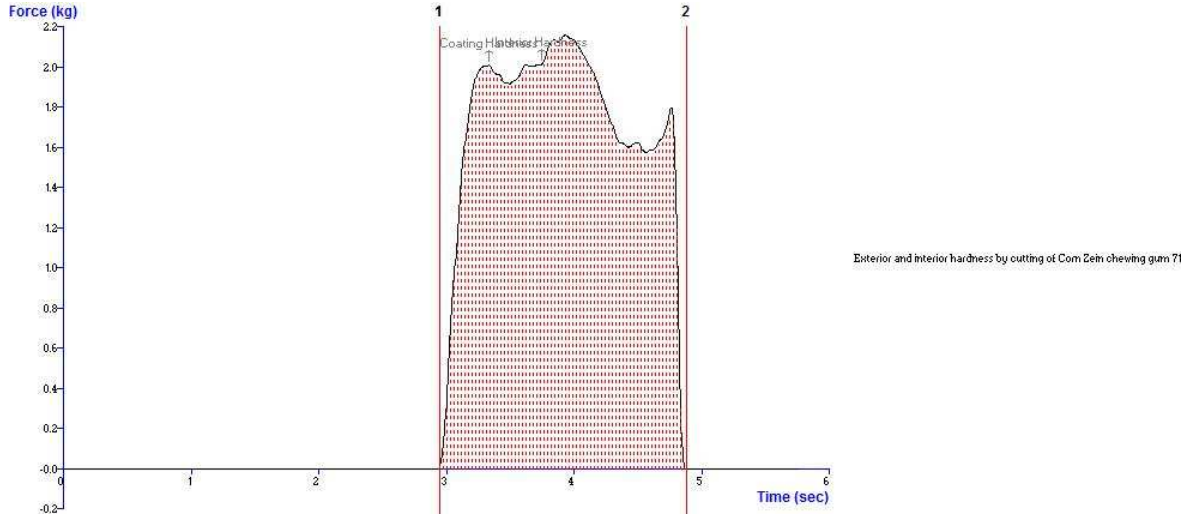

Fig 12: Exterior and interior hardness curve of MCG-7 corn zein gum formulation

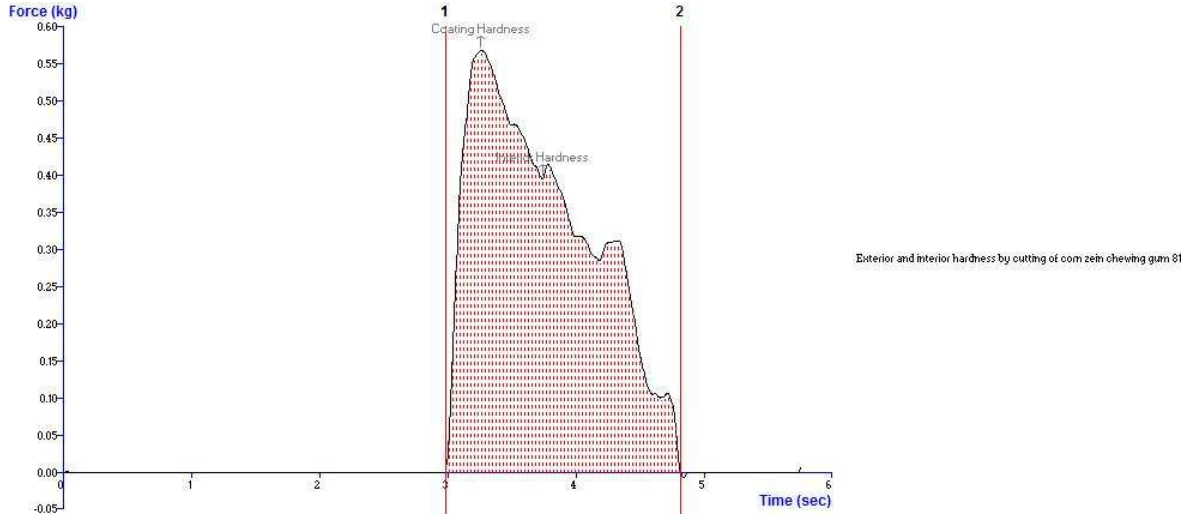

Fig 13:Exterior and interior hardness curve of MCG-8 corn zein gum formulation

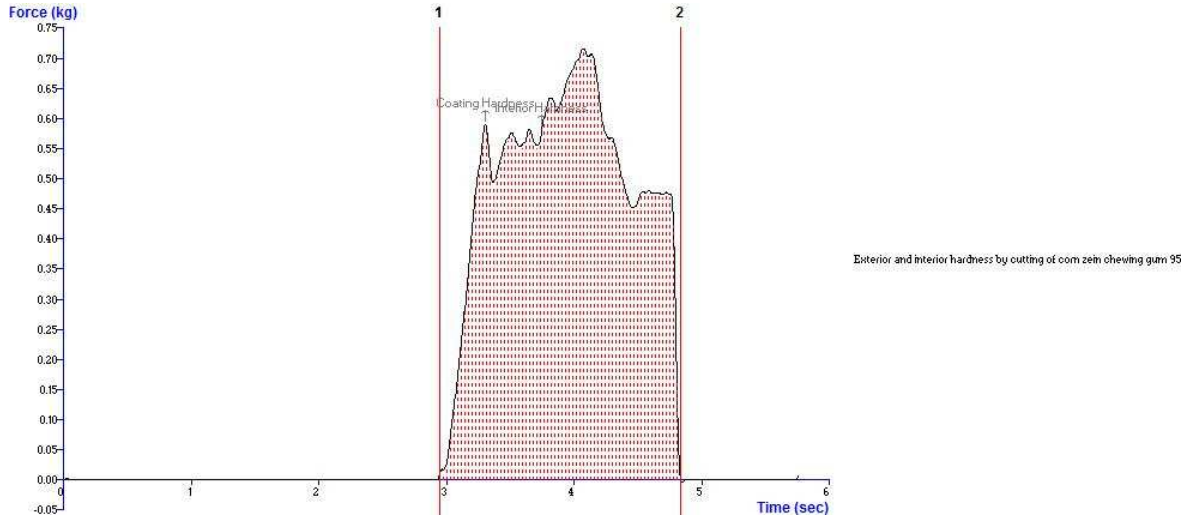

Fig 14:Exterior and interior hardness curve of MCG-9 corn zein gum formulation

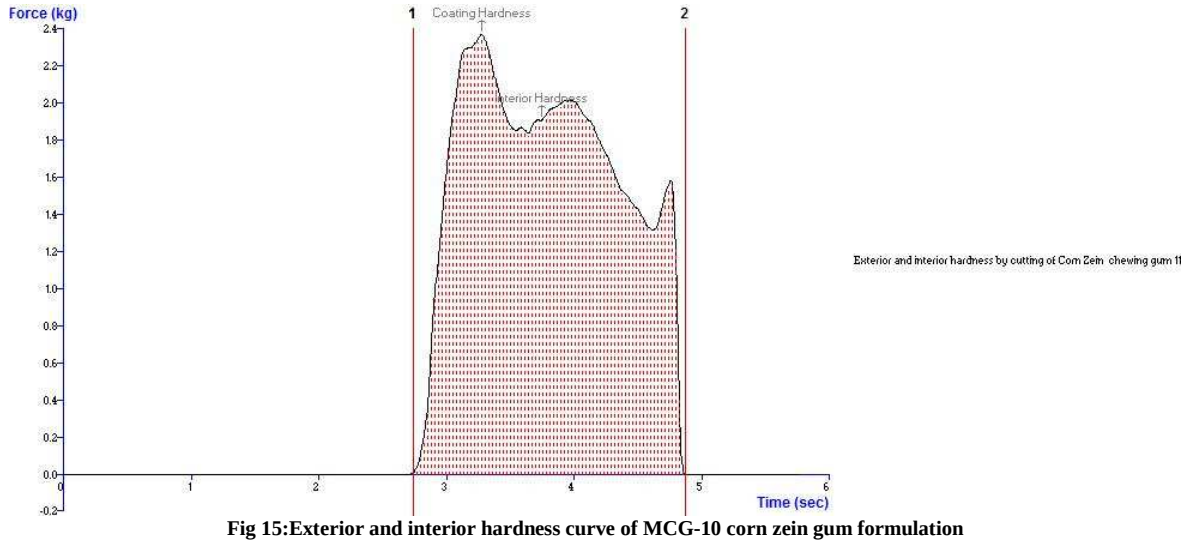

Fig 15:Exterior and interior hardness curve of MCG-10 corn zein gum formulation

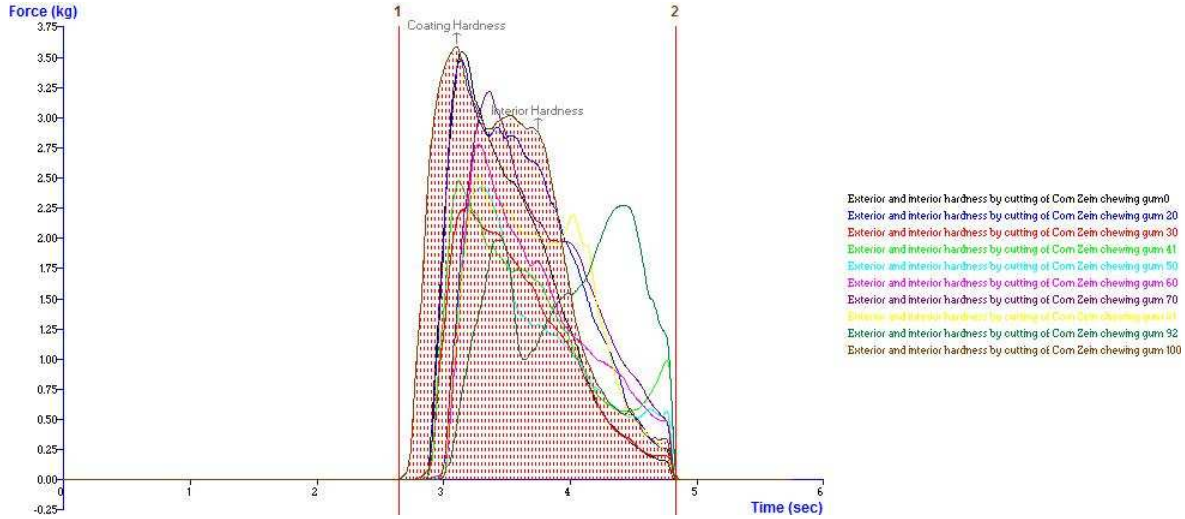

Fig 16:Exterior and interior hardness overlap curve of (MCG-1-MCG-10) corn zein gum formulation.

Table 7:Hardness obtained by coating 1.

Test ID	Batch		Coating Hardness Kilograms Kg	Interior Hardness Kilograms Kg	Area F-T 1:2,kg.sec
Start Batch 1	1				
Hardness by cutting of corn zein gum,MCG-1	1		1.755	0.501	1.985
Hardness by cutting of corn zein gum,MCG-2	1		1.008	0.357	1.526
Hardness by cutting of corn zein gum,MCG-3	1		1.109	0.680	1.984
Hardness by cutting of corn zein gum,MCG-4	1		1.848	0.336	1.686
Hardness by cutting of corn zein gum,MCG-5	1		1.038	0.281	1.373
Hardness by cutting of corn zein gum,MCG-6	1		1.017	0.324	1.093
Hardness by cutting of corn zein gu ,MCG-7	1		0.858	0.690	1.822
Hardness by cutting of corn zein gum,MCG-8	1		1.552	0.227	1.198
Hardness by cutting of corn zein gum,MCG-9	1		1.305	0.919	2.036
Hardness by cutting of cornzeingum,MCG-10	1		0.739	0.596	1.512
Average:	1(F)		1.223	0.491	1.622
S.D.	1(F)		0.379	0.224	0.337
Coef.of variation	1(F)	STDEV("BATCH")/AVERAGE("BATCH")*100	30.991	45.772	20.007

CONCLUSION

Effect of different formulations of chewing gum on final product's textural characteristics was successfully shown by Texture Analyser.

The Texture Analyzer does not require in-depth rheological training of the operator, either to run the test or interpret the results, which makes it very suitable for use in research & development. Using the same data results can be calculated repeatedly as per the convenience of the operator without repetition of tests. Testing with this instrument is ideally suited to product development or product standardization.

Though chewing gum as a drug delivery system has gained wide acceptance only within smoking cessation and oral health care, clinical trials have proven that there are therapeutic advantages to be gained by using chewing gum as a drug delivery system through exploiting the effects achieved by chewing gum per se, the convenience of the delivery system, and the possibilities of having buccal absorption or local effect of an active substance. Furthermore, one of the trials has indicated that chewing gum as drug delivery systems are possibly safer for active substances that are susceptible to abuse. Chewing gum formulations may also be less prone to accidental overdose.

Corn-zein chewing gum samples were coated and these coatings are brittle in nature and thus shown as fracturability in the results obtained. Different values of fracturability (of coatings) are observed in the graphs.

Above studies show that all the parameters obtained by texture analysis by Texture Analyser from SMS, UK can be complemented by the sensory evaluation data.

This study demonstrated the feasibility of using corn zein as a gum base and its potential for future optimization. Corn zein samples included in this study showed the potential for future optimization. The formulation containing triacetin demonstrating its desirable textural characteristics.

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