Textural and Physical Properties of North American Stick Margarines

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Compression of cylindrical samples was found to be a sensitive method in detecting differences in textural attributes in stick margarines. Constant speed penetration by a probe was the second most sensitive method, while the American Oil Chemists' Society (AOCS) cone penetrometer was the least sensitive method. Canola margarines were significantly harder than soybean margarines. Solid fat content in the product is related to its texture only to a certain extent. The nature of the crystal network is also of importance. To estimate the solids in the final product by pulsed nuclear magnetic resonance it is best to use the AOCS cooling method with a tempering step at 25°C. The International Union of Pure and Applied Chemistry (IUPAC) cooling method results in much higher values.

Texture has been defined as the external manifestation of structure and composition. Texture of margarines and butters usually includes properties such as hardness, spreadability, the presence of free moisture, graininess, brittleness, oiliness and stickiness (1). The products containing 80% fat can be considered plastic fats consisting mainly of crystals and liquid oil. The rheological properties are determined by a three-dimensional network of fat crystals. The forces that hold the three-dimensional network together consist of primary bonds, which result from crystals growing together (irreversible) and secondary bonds held together by weak London-van der Waals forces (reversible). The crystal network provides margarines and butters with visco-elastic behavior. The products behave like rigid solids until a deforming stress exceeds the yield value and the product starts to flow like a viscous liquid (2). Margarines differ in textural properties because of the strength of the crystal network, which is related to the amount of crystals present, the size of the crystals and strength of the primary and secondary bonds. The amount of crystals present is estimated by the solid fat index (SFI) in the fat by dilatometry, wideline or pulsed nuclear magnetic resonance (NMR). In the product itself it can be estimated by pulsed nuclear magnetic resonance (pNMR) (3). Margarines and butters containing relatively large crystals (>5 μm) at high solid content are harder, more brittle and grainy than those containing small crystals. At low solid content large crystals cannot incorporate as much liquid oil as small crystals, and the products become oily (4). Large, needle-like crystals are usually β crystals, while small ones are usually β' crystals. The strength of the primary bonds can greatly increase after manufacture. Transition of β' to β crystals is accelerated by temperature flucturations.

The most commonly used method for determining the textural properties of margarines is the cone penetrometer as described in AOCS method Cc16-60 (5). In this method the cone is driven into the product by the force of gravity and the penetration depth is measured. Hayakawa and deMan (6) suggested the term Hardness Index where the weight of the cone assembly is divided by the depth of penetration. Haighton (7) proposed the term yield value where the weight of the cone assembly is divided by the depth of penetration to the power 1.6. Vasic and deMan used a different formula to calculate the yield value (8). In constant speed penetration tests using different sizes of punches deMan (9) found a direct relationship between punch area and penetration force in the case of plastic fats. This relationship was later confirmed by Kamel and deMan (10). The stress values (g/cm^2) obtained with punches of different surface areas were not significantly different from each other, indicating that the shearing stress caused by the perimeter of the punch was negligible. Using foods of cellular structure, Bourne (11) found that the perimeter of the punch did influence the force of penetration. deMan *et al.* (3) suggested a hardness evaluation of margarines and shortenings by constant speed compression of cylindrical samples. Davis (12), experimenting with a lard and a shortening, recommended creep analyses for quality control purposes in the rheological properties of semi-solid foodstuffs. Pompei *et al.* (13) developed the Pompei Spreadability Cell, which is hooked up to an Intron Universal Testing Machine to evaluate the spreadability of butter and margarines.

This study was undertaken to evaluate the textural properties of North American stick margarines by three different methods. Textural results are compared with the solid content in the product. The solid content in the product is compared with that determined in the separated fat according to two official methods. Canadian margarines are usually manufactured from canola oil, while U.S. margarines are usually manufactured from soybean oil. Special attention was paid to these two categories. Three butters were also included for comparison.

MATERIALS AND METHODS

Samples were purchased from supermarkets in the provinces of Saskatchewan (Sas), Manitoba (Man) and Ontario (Ont) in Canada, and the states of California (Cal) and New York (N.Y.) in the United States of America. All samples were transported in coolers containing ice and stored in the refrigerator thereafter. The samples were coded with a letter and a number. Samples labeled with the same letter but with different numbers had the same brand name, but came from different areas.

The fat was obtained by melting part of the margarine in the oven. After removal of the water layer, the fat was dried and filtered. Prior to all textural evaluations, samples were conditioned at 10 or 21°C for 24 hr.

Constant weight penetration. The hardness of the samples at 10 and 21°C were measured with the same cone penetrometer, using AOCS method Cc 16-60 (5). The hardness index was calculated by dividing the mass of the cone assembly (= 92.5 g) by depth of penetration in mm (6). Yield value I was calculated according to the formula of Haighton (7) as follows: $Y = K\ W/p^n$ where

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Y = yield value, K = 19 for a 20° cone, W = 92.5 g (weight of cone assembly), p = penetration depth in 0.1 mm and n = 1.6 for margarines and shortenings. Yield value II was calculated according to the formula of Vasic and deMan (8) as follows: $Y = 10W/(.5625\ p^2 + 25.25\ p + 50.265)$. W and p are the same as in Haighton's formula. The yield values are usually expressed as kg/cm^2. In this paper they are expressed as N/cm^2 by multiplying the results by 9.807. An average of ten hardness measurements were made on each sample at both temperatures.

Constant speed penetration and compression. The Instron Universal Testing Machine (IUTM) and the Ottawa Texture Measuring System (OTMS(were used in both the penetration and compression tests. The OTMS was placed in a 10°C temperature controlled room for all 10°C measurements. In both instruments the load cells were hooked up to a strain-gage conditioner which was connected to an Apple //e computer by means of an A-D converter (3). Capacities of the load cells were 2.09, 4.55 or 50.0 kg, depending on the peak force and strength of the samples. Cross-head speed in all cases was 1.0 cm/min.

In texture measurements it is important not to disturb the integrity of the crystal network when sampling the material. Sampling in the constant speed penetration test was done by means of filling polypropylene stoppers (Bacti-Capall, Fisher Scientific, Cincinnati, OH) which had a hole drilled in the bottom. The cups were 25 mm high and 16 mm in diameter and were filled by pushing the cups into the margarines and butters. A stainless steel punch (30° angle; $A = 0.332\ cm^2$) was driven into the samples to a depth of 1.5 cm for the actual measurement.

In the compression test, cylindrical samples of 2 cm in height and 2 cm in diameter were compressed to 1 cm (50% compression) between flat plates. The samples were taken from the margarines and butters by means of a stainless steel tube with a close fitting plunger and were sized by a wire cutter.

In both tests four to five replicates were run. The force-deformation curve of each sample was recorded and was stored on floppy disks. ESRI software was used to analyze the curves (3). In order to compare the penetration test with that of the compression test, the average penetration values were divided by the area of the penetration punch ($A = 0.332\ cm^2$) and the average compression values by the area of the sample ($A = 3.14\ cm^2$).

Solid fat content. The Solid Fat Content (SFC) of the samples was measured by pNMR, using a Bruker PC/20 Series NMR analyzer (Minispec) (3). The SFC of the separated fats was determined using AOCS method Cd-57 (5) with tempering at 25°C, and the IUPAC method (14), which involves no tempering and where samples are left at 0°C for 1.5 hr. Solid fat content in the margarine samples was determined as previously described (3). The product samples were left in either a 10 or 21°C incubator for 24 hr. Prior to measurement by pNMR, these samples were conditioned in a waterbath for 30 min at the same temperatures.

Statistical analysis. The Wilcoxon-Mann-Whitney two sample test was used to determine significant differences between two rows of unpaired values. Wilcoxon's signed rank test was used to determine significant differences between paired values. Correlation between paired values was estimated by linear regression (15).

RESULTS AND DISCUSSION

Postmus *et al.* (16) previously analyzed the samples for their chemical composition, and according to those results the samples were divided into the categories displayed in Tables 1 and 2.

Figures 1 and 2 show the force-deformation curves of the penetration and compression tests of canola sample 8 and soybean sample 10, respectively. During the compression of the cylindrical samples a diagonal or a

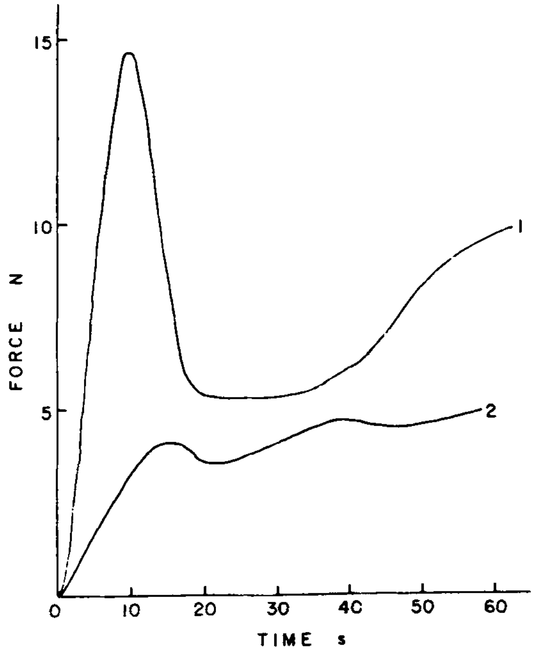

FIG. 1. Force-deformation curves of sample 8. (1) Compression of cylindrical sample. (2) Constant speed penetration of sample contained in small cup.

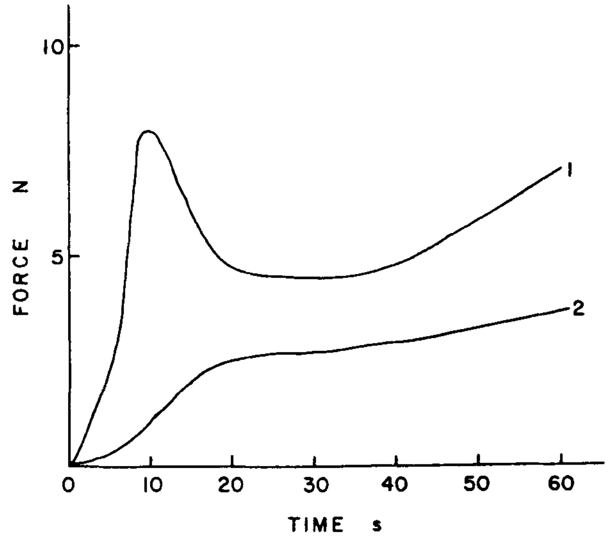

FIG. 2. Force-deformation curves of sample 10. (1) Compression of cylindrical sample. (2) Constant speed penetration contained in small cup.

TABLE 1

Textural Characteristics of Margarines and Butters at 10°C

		Constant speed		Constant weight		
Sample	Origin	Penetration force at 1.5 cm N/cm^2	Compression breaking force N/cm^2	Hardness index g/mm	Yield value I N/cm^2	Yield value II N/cm^2
Canola						
1 A1	Man.	8.1	3.3	9.3	11.0	1.1
2 A2	Sas.	NA	7.5	23.5	48.4	4.7
3 B1	Man.	19.1	5.5	18.1	31.9	3.2
4 B2	Sas.	18.8	6.0	19.7	36.4	3.6
5 C1	Sas.	15.9	4.3	16.6	27.8	2.8
6 D1	Man.	19.1	4.3	15.2	24.0	2.5
7 E1	Ont.	18.8	6.3	20.0	37.4	3.8
8 F1	Ont.	12.3	5.3	14.6	22.6	2.4
Average		16.0	5.3	17.1	29.9	3.0
Soybean						
9 G1	Cal.	10.5	2.8	14.3	21.9	2.3
10 G2	N.Y.	9.3	2.2	15.9	25.9	2.6
11 H1	Cal.	10.4	2.2	18.0	31.6	3.2
12 H2	N.Y.	11.0	2.2	17.3	29.6	3.0
13 I1	Cal.	11.7	2.5	15.4	24.6	2.5
14 J1	N.Y.	13.3	3.0	17.9	31.3	3.1
15 K1	N.Y.	11.1	2.4	13.4	19.7	2.1
16 L1	N.Y.	10.7	2.6	12.7	18.0	1.9
Average		11.0	2.5	15.6	25.3	2.6
Mixt. canola and soybean						
17 G3	Ont.	11.3	3.6	14.8	23.0	2.4
18 H3	Ont.	15.5	4.1	14.4	22.1	2.3
19 J2	Ont.	9.5	1.8	10.7	13.7	1.4
Corn or sunflower						
20 M1[a]	Ont.	8.5	2.1	10.1	12.6	1.3
21 N1[a]	N.Y.	9.3	2.0	10.5	13.3	1.4
22 O1[b]	Cal.	7.6	1.8	11.7	17.2	1.8
Mixt. vegetable and animal						
23 P1[c]	N.Y.	24.7	7.8	28.9	67.4	6.4
24 Q1[d]	N.Y.	41.3	10.5	40.2	114.2	9.8
Butter						
25 R1	Ont.	48.4	16.3	43.8	131.4	11.8
26 S1	Ont.	35.3	7.1	25.8	55.9	5.4
27 T1	Ont.	33.3	11.0	29.9	71.2	6.7

[a]Corn. [b]Sunflower and soybean. [c]Butter and corn. [d]Beef and cotton seed.

TABLE 2

Mean Coefficients of Variation (C.V.) of the Replicates of Textural Evaluation Using Three Textural Evaluation Methods at 10°C

Method	C.V. %
Cone penetrometer	6.6
Constant speed penetration	5.3
Constant speed compression	10.7

V-shaped crack developed in the softer samples. In the harder samples many cracks appeared or the fat cylinder fell apart. The latter case is illustrated in Figure 1 by the sharp downward slope of curve 1. The computer program computed the slope of the initial curve—which is called firmness—the peak force and the energy of penetration, which is the area under the curve. Correlation between firmness, energy and peak force in the penetration test was .99. Because of this excellent correlation only the peak force values are reported since on most texture instruments peak forces are displayed automatically. In the compression test the first peak is considered. Some samples showed a second peak which was higher than the first peak. The first peak represents the force necessary to break down the crystal network.

Table 1 shows the textural characteristics of the samples. There was no significant difference in hardness index values or in yield values between the canola and soybean margarines. However, there was a significant difference ($P < 0.01$) in the compression peak forces between the two kinds of margarines. The compression peak forces for canola margarines were, on the average, twice as high as those for soybean margarines. Penetration peak forces were also higher for canola margarines than for soybean margarines. When samples are very brittle the penetration test tends to give low values because the punch breaks off small pieces of the sample during the test. This is illustrated in curve 2 of Figure 1 (sample 8). The horizontal part of the curve shows dips which indicate breaks in the sample. Curve 2 of Figure 2 (sample 10) does not show this. The cone penetrometer did not show a difference in hardness index between these two samples. The corn/sunflower margarines showed the lowest values for all of the three textural attributes. The vegetable-animal mixtures nos. 23 and 24 (Table 1) were much harder than the vegetable margarines. Butter is processed in a different way than margarine. A considerable amount of globular fat is still present in continuously churned butter. Butter no. 25 was prepared by the Golden Flow process (17), and this process resembles a margarine process. Postmus *et al.* (16) showed photomicrographs of no. 26 and 27 to have typical globular crystals, whereas the crystals in no. 25 were coarse and needle-like. The latter crystals produce a tighter network. Although the penetration forces for no. 26 and 27 were high (Table 1), the compression forces were relatively low, indicating the butters to have a different structure than margarine. In the compression test at 10°C, all samples showed a peak at the beginning of the test. Some peaks were sharper than others. The sharpness or broadness of the peak may be an indication of brittleness. In butter samples 26 and 27 these peaks were broad, whereas in butter sample 25 the peak was sharp.

Yield values (N/cm^2) calculated according to Haighton's formula were the highest in all cases (Table 1). Yield values as calculated by the Vasic and deMan formula are very close to the breaking forces in the constant speed compression tests. Values of the breaking forces have a wider range than the yield values II, indicating that the compression test is more sensitive in detecting differences between samples. Haighton (7) states that yield values and viscosity are closely related. The peak forces in the compression tests were forces that broke or crumbled the samples. In the constant speed penetration test small cups were used. When the probe penetrated into the margarine a kind of back extrusion took place. Values obtained in this test will therefore partly reflect the viscous behavior of the samples. Penetration depth by the cone penetrometer ranged from 9.9 to 2.1 mm or from 99 to 21 scale units. Hayakawa and deMan (6) stated that the most desirable range of penetration values is from 15 to 150 units. Haighton indicated that margarines of yield values from 8 to 10 N/cm^2 (800–1000 g/cm^2) are hard but satisfactorily spreadable. Those of values from 10 to 15 N/cm^2 are too hard and at the limit of spreadability. None of the samples tested would qualify as being spreadable at 10°C. Compression tests at 21°C showed a peak at the beginning of the test only in samples 2, 7 and 8. In the rest of the samples the compression curve showed a gradual increase in force with or without a plateau force in between. Data on texture at 21°C are not supplied as they were less reproducible because of the softness of the product. This was especially the case with the cone penetrometer.

The coefficient of variation (C.V.) for the textural methods was calculated from the standard deviation (S.D.) of the replicates of each sample. The mean C.V. was then calculated for each of the textural methods from the C.V. of all samples. The results are shown in Table 2. Compression tests gave a higher C.V. because the standard deviation (S.D.) of the harder samples was greater than those of the softer ones. The crystals of canola samples 1, 2, 7 and 8 were in the β form (16), all other margarines were in the β' form. Crystals as observed by the polarizing microscope were needle-like for all β margarines, and were very small for the corn margarine. The crystals of the soybean margarines were generally smaller than those of the β' canola margarines. Canola margarines require the incorporation of palm oil to stabilize the β' crystallinity. No palm oil was incorporated in sample nos. 7 and 8. Although palm oil was incorporated in nos. 1 and 2, the amount was insufficient to prevent the formation of β crystals. Table 3 shows the pNMR results of the following: SFC of product, difference between SFC of fat by the IUPAC cooling method and SFC of product, and difference between SFC of fat by the AOCS cooling method with tempering at 25°C and SFC of product. Table 3 shows that the IUPAC cooling method gives higher results than the actual product at both 10 and 21°C. Results using the AOCS cooling method are also higher than those products at 10°C, but to a lesser extent. The exceptions are nos. 2 and 7 where the solids are higher in the product than those obtained by using the AOCS cooling method. Both samples showed β crystallinity and high compression and hardness values (Table 1). At 21°C there was no difference in SFC between the results obtained with the AOCS cooling method and those measured in the actual product. Table 4 shows the mean differences between the SFC values. The linear regression equations and the correlation coefficients are displayed in Table 5. There was a poor correlation between the SFC of the product and that of the separated fats. This is not surprising since processing conditions and, to a certain extent, storage conditions greatly influence the solid content in the final product. The AOCS cooling method provides closer estimates of solids in the final product. Correlation coefficients between the various textural methods and the SFC are displayed in Table 6. The correlation within the textural methods was good (Table 6), while the correlation between the texture methods and the SFC was poor. This means that the amount of crystals (or solids) in a product does not fully determine the texture. The nature of the crystal network is also of importance. The polymorphic form of the crystals also plays a role in the strength of the crystal network. In addition, large crystals may result in a sandy mouth feel (4).

All three texture methods estimate some aspects of the strength of the crystal network. The ranges of values in the actual tests were as follows: cone penetrometer 21–99 scale units, constant speed penetration 2.5–16 N and constant speed compression 5.6–51 N. In the compression test canola margarines were significantly harder than soybean margarines. Canola margarines showed a wide range in textural characteristics, whereas soybean margarines were more uniform. An advantage in using the small cups in the constant speed penetrometry is that large commercial blocks of margarines can be sampled more conveniently. Constant speed penetrometry using the probe of

TABLE 3

SFC of Product, Difference Between SFC of Fat Using IUPAC Cooling Method and SFC of Product, and Difference Between SFC of Fat Using AOCS Cooling and Tempering at 25°C and SFC of Product (%)

		10°C			21°C		
Sample	Origin	Product	IUPAC-product	AOCS-product	Product	IUPAC-product	AOCS-product
Canola							
1 A1	Man.	30.4	16.8	3.5	19.6	1.1	−3.7
2 A2	Sas.	35.6	13.4	−2.0	17.8	3.4	−1.1
3 B1	Man.	28.7	19.9	6.4	17.1	3.1	−.5
4 B2	Sas.	26.8	19.9	5.5	14.1	5.7	1.2
5 C1	Sas.	29.9	18.0	5.0	15.2	4.7	1.4
6 D1	Man.	31.0	14.9	.7	16.8	.5	−1.4
7 E1	Ont.	34.7	6.0	−3.4	16.6	.5	−1.9
8 F1	Ont.	23.5	15.1	6.9	16.0	−.4	−2.8
Soybean							
9 G1	Cal.	22.3	13.6	4.8	13.5	2.6	.1
10 G2	N.Y.	22.8	10.8	8.8	14.0	3.8	.2
11 H1	Cal.	31.1	10.1	1.7	15.4	2.9	−.1
12 H2	N.Y.	25.4	11.4	4.3	17.1	4.5	−.1
13 I1	Cal.	22.2	15.5	7.3	15.5	5.1	2.4
14 J1	N.Y.	25.7	14.0	4.9	18.0	3.7	−.2
15 K1	N.Y.	23.6	10.6	3.0	15.2	5.2	−.1
16 L1	N.Y.	23.4	9.5	1.9	16.1	6.0	.4
Mixt. canola and soybean							
17 G3	Ont.	30.3	15.3	5.6	15.2	2.2	−.9
18 H3	Ont.	32.3	8.4	1.0	16.0	4.4	.8
19 J2	Ont.	26.7	12.2	2.9	16.0	2.6	−.6
Corn or sunflower							
20 M1[a]	Ont.	21.9	13.2	5.8	14.3	2.5	.5
21 N1[a]	N.Y.	22.8	6.1	.2	14.1	5.0	−.4
22 O1	Cal.	19.8	11.1	4.8	12.3	3.3	1.8
Mixt. vegetable and animal							
23 P1[c]	N.Y.	24.3	13.7	2.3	11.8	4.3	0
24 Q1[d]	N.Y.	29.2	7.9	3.6	15.2	1.4	−2.5
Butters							
25 R1	Ont.	34.8	11.5	2.0	12.4	2.2	.1
26 S1	Ont.	35.0	9.6	.4	14.1	.3	−1.9
27 T1	Ont.	37.6	10.3	.3	15.6	−.3	−2.0

[a]Corn. [b]Sunflower and soybean. [c]Butter and corn. [d]Beef and Cottonseed.

TABLE 4

Mean Differences Between the Solid Fat Content of the Fat Determined by pNMR and Two Tempering Methods (IUPAC and AOCS) and the Solid Fat Content of the Margarine or Butter as Determined on the Product Directly by pNMR

Method	10°C	21°C
IUPAC-product	12.6	3.1
AOCS-product	5.1	−.6

TABLE 5

Linear Correlation Between the SFC of the Products and/or Their Separated Fats (n = 27)

Parameters		10°C[a]		21°C[b]	
x	y	Cor. coef.	Equation	Cor. coef.	Equation
Product	IUPAC	0.79	Y = 0.92X + 14.77	0.65	Y = 0.86X + 5.27
Product	AOCS	0.85	Y = 0.676X + 12.09	0.67	Y = 0.64X + 5.13
AOCS	IUPAC	0.91	Y = 1.34X − 0.94	0.89	Y = 1.22 + 0.23

[a]Solid content range from 20 to 28. [b]Solid content range from 10 to 20.

TABLE 6

Correlation Coefficients Within Textural Methods and Between Textural Methods and SFC at 10°C

Method	N	r
Peak force penetration and peak force compression	26	.91
Peak force penetration and hardness index	26	.95
Peak force compression and hardness index	27	.93
Peak force penetration and SFC product	26	.63
Peak force compression and SFC product	27	.63
Hardness index and SFC product	27	.53
Peak force penetration and AOCS	26	.57
Peak force compression and AOCS	27	.63

area 0.332 cm^2 is not suitable for hard margarines. Smaller area probes may be more suitable. Haighton's yield values are about ten times as high as those of Vasic and deMan (8). Hayakawa and deMan (18) also found a ten-fold difference between the two calculated yield values. Yield values II are of the same magnitude as the breaking forces, and therefore the breaking forces represent the yield forces in the case of margarines at 10°C. Penetration forces are related partly to the viscous flow of the product. In order to estimate the solids in the final product of a margarine formulation it is best to use the AOCS method with a tempering step at 25°C. Solid fat content in a product is related to its texture only to a certain extent. Shortenings are manufactured differently than margarines. In addition, shortenings are tempered at 27–30°C and stored at room temperatures.

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