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The Addition of NaCMC
as a Anti-Redeposition Agent
in the Floatation De-inking
of Newsprint

by

Ronald R. Van Biervliet

A Thesis Submitted in
Partial Fulfillment of the
Course Requirements for
The Bachelor of Science Degree

WESTERN MICHIGAN UNIVERSITY
KALAMAZOO, MICHIGAN
APRIL 8, 1980

ABSTRACT

A study was performed to determine the effectiveness of NaCMC, as an anti-redeposition agent on newsprint, in a floatation deinking system. NaCMC performs the function of preventing soils from redepositing in many commercial laundry detergents. The study used six different surfactants; five were nonionic, and one was anionic. The effect of NaCMC was studied at three different levels of addition; 0.05%, 1.0%, and 5.0%. One set of hand-sheets was produced with peroxide for comparison purposes. The effect of pH at 9.0, 10.0, and 11.0 with NaCMC was also studied. The data indicates that NaCMC does act as a redeposition agent in nonionic floatation systems. The strength as measured by mullen significantly increased at 5.0% level of NaCMC addition. NaCMC does not appear to be compatible with anionic surfactants and NaCMC does not significantly increase brightness of the deinked pulp.

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THE ADDITION OF NaCMC IN THE FLOATATION DRINKING OF NEWSPRINT

Introduction

What is NaCMC

Sodium Carboxy Methyl Cellulose (NaCMC), also called sodium cellulose glycolate, is a chemical derivative of alpha cellulose. NaCMC was first developed in Germany during World War I, patents having been taken out by E. Jansen on behalf of Deutsche Celluloid Fabrik in 1918¹. NaCMC is made by steeping cellulose derived from wood pulp or cotton linters in sodium hydroxide solution and treating the resulting alkaline mass with sodium monochloracetate or with chloracetic acid² (figure 1). CMC may be produced in either batch process or a continuous process. A variety of grades are marketed. The two major differences between grades of NaCMC are the chain length of the cellulose and the degree of substitution. The degree of substitution refers to the number of groups that may be substituted on a glucose ring. The largest number of substitutions in theory that may be made on a glucose ring are three (figure 2). But in practice this is not true due to the severity of the conditions that would be necessary in order to obtain complete substitution.

The degree of substitution is controlled by the time the alpha cellulose is allowed to react with sodium monochlor-acetate or chloracetic acid and the temperature of the reaction medium.

The chain length of the cellulose is controlled by the period of

of time the alpha cellulose is allowed to react with alkali and the mechanical action that occurs after the alkali reaction. When purchasing NaCMC, the degree of substitution is usually based on ten glucose units; example, substitution = 7 or 7 of 30 hydroxyl groups had been substituted with the carbon-methyl group. The viscosity of the NaCMC is typically controlled by the cellulose chain length, the longer the chain the higher the viscosity and is classified high, medium, or low viscosity (Table I). The principle by-products when producing NaCMC are sodium chloride, sodium glycolate, residual sodium bicarbonate and sodium carbonate. These compounds are usually all present in technical grades of NaCMC.

How NaCMC Relates to Drinking

One of the unusual properties of NaCMC is its ability to prevent redeposition of soil (ink) that has been removed from fabrics (cellulose fiber) by use of built soaps (surfactants)³. Another property of NaCMC is the ability to form films that are resistant to greases and waxes. There are two theories as to why this occurs. The first theory states that CMC forms a cloud around the soil (ink) particles preventing redeposition to the cellulose. The second theory states that CMC forms Van der Waals linkages between the hydroxyl groups on the CMC due to the close geometrical configuration of the molecules. These linkages to the cellulose ring increase the negativity of the combined compound preventing redeposition of the soil. The second has become the most excepted due to experimental indications. From theoretical considerations,

properties of CMC might be caused by electrostatic repulsion between negatively charged dirt particles and increased negativity of the cellulose induced by the carboxy methyl groups of adsorbed CMC⁴. The common detergent grade NaCMC is a medium viscosity with a substitution of 7.5. A higher substitution CMC is not used because, although increasing the substitution, will increase the negativity of the charged molecule; it also decreases the affinity of the CMC to attract itself to cellulose. The phenomenon occurred because there are fewer hydroxyls available for hydrogen bonding to the cellulose. It appears that in the first stage of washing, the surface active ingredients of the detergent cause the greasy material that coats such fiber of the soiled fabric to roll up into globules which during rubbing or shaking are detached from the fabric and float off into the surrounding liquid. The globules take the form of an emulsion or a dispersion (if solid), but as both forms are relatively unstable, there would be continuous redeposition of soil onto the surface of the fabric if it were not for the presence of the phosphate, silicates, and of the soil suspending agent⁵. NaCMC may be utilized in the floatation de-inking of newsprint as a redeposition agent. Due to the close physical and chemical similarities between cotton fabric and cellulose in paper, the general reactions that occur in washing of fabric should hold true in the de-inking of newsprint. One of the major problems with the de-inking of newsprint is that the resulting pulp is low in strength and brightness. The strength and brightness decrease may occur due to the ink particles

redepositing on the cellulose fibers and interfering with hydrogen bonding between the cellulose chains. The brightness can be artificially increased by the addition of peroxide, but this process is expensive. It would be realistically impossible to expect all the ink to be released during the floatation process. The assumption is that some of the ink particles that are released during the drinking process do redeposit on the cellulose before being removed from the system. Preventing redeposition would yield higher strength and brightness values than is obtained with standard newsprint de-inking formulations currently used in industry.

Chemical and Physical Nature of the Floatation De-inking System

What is Floatation De-inking

Floatation de-inking is a specific process for removing ink particles present in wastepaper. A physico-chemical environment is utilized to remove contaminants. A floatation de-inking system will yield 85-95% of the raw material as final product which is significantly higher than other types of cleaning systems.

Floatation consists of introducing chemicals to the raw stock that will separate the ink particles from the fibers and promote the attraction between ink particles and air bubbles. The chemical additives used in the process are alkalis, soaps, dispersants and bleaching agents. The mechanics of floatation consists of producing sufficient turbulence to separate the ink particles from the fibers. Next, to produce bubbles of proper dimension to act as vehicles to float contaminants to the surface.

Function of Surfactant and Bubble Formation

Foam is one of eight classes of colloidal systems; namely, a gas dispersed in a liquid. In a floatation de-inking system the gas is air and the liquid is water. The presence of gas and a pure liquid is not alone, sufficient to produce foam, because pure liquids do not foam. A third component is required. This component, called a surfactant, is soluble in the liquid. Surfactants are chemicals composed of molecules having both water soluble and water insoluble portions within the same molecule.

These molecules will concentrate at the air water interface in order to satisfy both portions of the molecule. The hydrophobic end attaches itself to the air bubble. Once the ink particle has attached itself to the air-surfactant bubble, it is raised to the surface and removed by mechanical action. Surfactants are typically long chain hydrocarbons or alkyl (aryl) halides (triton x-100 is a octyl phenoxy polyethoxy ethanol) that possess enough hydroxyl groups to attract the air bubble. From the existing experiments the combined use of nonionics and alkalis must be considered as the most satisfactory method of detaching printing inks⁷.

Air bubble size is very important in the floatation system. Due to the fluid dynamics of air in water under pressure, bubbles will increase as they rise to the surface. If the diameter of the air bubble becomes too large, the surface tension of the film surrounding the air bubble becomes too great, and the bubble will burst releasing contaminants back into the pulp. Temperature has a marked effect on the surface tension of the slurry. At low temperatures air will remain dissolved in the liquid and fewer bubbles will be formed. At high temperatures air will not dissolve in the liquid but will be present in the gaseous form (bubbles) only. Bubble formation is also decreased at the high and low end of the scale of the pH range. The quantity of air required for normal floatation is very small; approximately 10% and above 30% (based on volume of liquid present at 1% consistency) is unfavorable in floatation⁸. For efficient floatation de-inking

to take place, there must be sufficient amount of turbulence to disperse the bubbles uniformly throughout the cell.

Calcium chloride may also be added to the system. Calcium chloride is used to harden the water to promote ideal conditions for bubble formation during the floatation stage. If the water is too soft (not enough ions present), ideal bubble formation will not occur.

How Surfactants and Bubble Formation Effect the Addition of NaCMC

Typically, there are three classes of surfactants; anionic, cationic, and nonionic. In a chemical system where a high concentration of anionic molecules (CMC^-) are present, the probability of the surfactant floating a significant percentage of the CMC-fiber to the surface is great. Therefore, the use of a cationic surfactant must be eliminated from the system. The second selection could be the use of an anionic surfactant. Unfortunately, anionic surfactants tend to precipitate out under hard water conditions. Kalamazoo water is quite hard (250-400 ppm as CaCO_3)⁹. Therefore, anionic surfactants must be eliminated from our choices. Nonionic surfactants offer many benefits to our system. Unlike anionic or cationic surfactant, nonionics do not react with either the water hardness or the NaCMC. High concentration of surfactant decreases the strength of de-inked newsprint. The surfactant (dispersant)

distributes a proportion of the ink particles too finely in the system, under 5 mu, which is one of the reasons that ink particles are not completely removed from the system. Particularly, in newsprint where no binder is used to hold the carbon particles together. These vehicles consist totally of hydrocarbons and are not subject to saponification with alkali. Removal of these materials must depend on emulsification and/or mechanical removal¹⁰ (table II).

Role of Silicates and Peroxide

Sodium silicate (NaSiO_3), soda ash (Na_2CO_3) or sodium hydroxide is added to the system to raise the pH of the slurry raw stock to 9-10. Silicates will react with binders used in some printing inks to saponify and release the ink particles. Saponification is a chemical reaction in which fatty acids react with alkali to produce soap. The alkaline conditions will also increase the reactivity of cellulose to hydrolysis. In a slurry composed of groundwood (newsprint) alkaline conditions above a pH 10 will tend to cause yellowing of the fiber. This is due to a reaction occurring between the lignin and the alkali. Under high pH conditions lignin will degrade primarily to a lower oxide state which produces the undesirable color. One of the unique advantages of sodium silicate is its ability to buffer at a pH close to 10. Sodium peroxide does not help to clean the pulp, but bleaches the cellulose. Sodium peroxide oxidizes the non-carbohydrates present, acting as a single stage bleaching sequence. This chemical sequence described is only an average.

Experimental Design

On the basis of the literature review the experimental design will have three variables:

- (1) pH
- (2) surfactant
- (3) NaCMC

A standard newsprint will be obtained printed by an offset printing method which is becoming the predominant method used by newspaper publication companies. The objective will be to obtain, first a sample of unprinted groundwood and samples of newsprint from the same source, the Western Herald. The first section of the experiment will be to re-slurry the groundwood (blank newsprint) and make handsheets to determine dirt count, brightness, whiteness, mullen, and tensile. The data will be used for comparison purposes for the rest of the experiment. Next will be to run successive trials on the newsprint according to table III. After determining the best formulation from the preliminary trials an effort will be made to fine tune the formulation and an economic study will be performed to determine if the formulation is cost effective.

Procedure

The following procedure was used in preparing the samples for testing:

- (1) Adjust 1.5 liters of tap water to the desired pH with sodium silicate.
- (2) Add 45 gms of O.D. fiber to the adjusted tap water.

- (3) Allow to stand for 15 minutes.
- (4) Slurry stock in an Industrial Waring Blender for 90 seconds at low speeds.
- (5) Add NaCMC (if required) and allow to stand in a constant temperature bath for one hour.
- (6) De-ink stock for 30 minutes in a Voith Lab Cell after adding surfactant.
- (7) Make three handsheets @2.5 gms for testing without the addition of any more dilution water on a Noble & Wood sheet machine.
- (8) Test handsheets for brightness, whiteness, tensile, and Mullen (Table III).

Discussion and Observation of Results

The dirt count (figure 3) in all cases decreased with the addition of NaCMC at the 5% level of addition. The addition of NaCMC was not a linear relationship with dirt count. At lower addition levels (0.05%), NaCMC was detrimental to the system; that is in three of the six surfactants used, NaCMC caused an increase in the amount of dirt specks in the sheets. The dirt specks on the handsheets, floatation de-inked with the anionic surfactant, steol CS-460, observed very large ink specks in comparison to the specks on the other handsheets produced. This observation indicates that NaCMC is not compatible with anionic surfactants. The surfactant Bio Soft-LD 95 is a surfactant produced by Stepan Co. and the chemical formulation is proprietary. Bio Soft LD-95 had a lower dirt count than the surfactant Triton X-100.

The brightness data of the handsheets was hard to interpret. In three of the samples tested (figure 4), there was no change in brightness with the addition of NaCMC indicating that decreasing the number of dirt specks does not measurably affect the brightness value. In two of the sets of handsheets, the surfactants Bio Soft LD-95 and Triton N-101, the brightness value increased with increasing % NaCMC. The increase in brightness was 48.5 to 50.2 indicating that brightness improved with increasing % NaCMC. In the Wa-Extra surfactant the brightness decreased at the 0.05% level of addition and increased at the 5.0% level of addition to a higher value of brightness than the 0% level of NaCMC. Indicating that at some level of addition between 0.05% and 1.0%, NaCMC can be a detriment to the system. This assumption was also indicated by the dirt count data. In generalizing the effect of NaCMC on brightness, in non-ionic floatation systems the addition of NaCMC will not measurably improve the brightness of the resulting pulp.

Whiteness data (figure 5) was tabulated to get some indication or cross reference between brightness and color of the actual handsheets. In three sets of samples tested, the whiteness value increased with increasing % NaCMC, indicating that NaCMC does improve the whiteness of the handsheets. In three sets of trials the whiteness values decreased with increasing NaCMC, indicating that NaCMC is detrimental to whiteness. The three sets of samples that showed no measureable change in brightness with increasing % NaCMC were the samples that showed a decrease in whiteness with increasing % NaCMC. Whiteness may be a more sensitive test to

show the effect of addition of NaCMC on the color of the handsheets.

In all cases mullen improved significantly with increasing NaCMC (figure 6). In three trials the mullen increased measureably at the 0.05% level of addition, indicating that even very small levels of NaCMC improve the mullen measureably. The three sets of samples that showed significant increase at the 0.05% level of addition of CMC showed decrease at the 1.0% level of addition. Indicating, some level of addition between 0.05% and 1.0%, the addition of CMC can be detrimental to the system. In three sets of samples the addition of CMC increased mullen linearly. Note that in all cases the addition of CMC improved the overall mullen of the handsheets possibly by increasing the crosslinking that occurred during sheet formation. NaCMC is known to be a strengthening agent. Some mention of sheet formation should be made at this point; in the procedure no dilution water was added to prevent washing out of the ink specks. This procedure caused poor sheet formation, which ultimately affects the overall strength of the handsheets, providing incorrect mullen values. But there is strong indication that improving mullen does occur with increasing % CMC.

The tensile data (figure 7) was also quite scattered. It is believed that most of the error in the tensile samples can be

explained by the formation of the handsheets. The tensile procedure only tests the weakest points of the sample and the sheet formation was non-uniform, explaining the inaccuracy of the tensile data.

The Effect of pH on NaCMC

The dirt count is significantly less (figure 8) approximately 12.5%, at a pH 10 then the pH levels of 9.0 and 11.0. Brightness (figure 9) increased with higher pH levels; the highest brightness value at pH 11.0; 49.4. The lowest value of brightness was obtained at the pH 9.0, 48.0. A comparative value was obtained at pH 10.0, 49.2, as compared to pH 11.0, 49.4. The whiteness data (figure 10) showed the pH 10.0 level had the highest value, 57.8, 3.0% higher than the pH 9.0, but only 1.04% higher than the pH 11.0. The highest mullen value (figure 11) was obtained at the pH 10 level, 17.3; 11.6% higher than the value obtained at pH 9.0 and 3.47% higher than the pH 11.0 level. Tensile (figure 12) steadily decreased with increasing pH, the highest value for tensile was obtained at the pH 9.0 level; 21.2% higher than the pH 11.0 and 13.5% higher than the pH 10.0 level. These differences in pH level may be by non-uniformity across the sheet surface. The above pH information indicates that the pH level of 10.0 is the best level in the overall evaluation.

The Effect of Peroxide

The addition of peroxide increased the number of dirt specks in

in the sheets produced, indicating that peroxide may have an attraction for inks. The brightness obtained for the pulp treated with peroxide, indicated that peroxide does significantly increase the brightness value, approximately 3.37% over the newstock de-inked at the same pH level and without peroxide. The whiteness value obtained was also higher than the value obtained at the pH 11.0 without peroxide, approximately 3.37% equivalent to the brightness difference obtained. The addition of peroxide improved the results of mullen over that of the sheets made without peroxide, but not greater than the value obtained with NaCMC (5.0%) at the pH 10 level, a value 8.67% higher was obtained. Indicating that NaCMC may act as a strengthening agent. The tensile value (peroxide) obtained was higher than any value for tensile without peroxide. This observation could be primarily due to non-uniformity in the sheet surface.

Conclusions

The mullen values had a significant increase in value approximately 11.8% indicating that NaCMC does act as a strengthening agent at the higher addition levels (5.0%). Dirt speck count decreased or remained constant in all of the surfactant tests performed. There appears to be a level of addition between 0.05% and 1.0% that may actually be detrimental to the system, as indicated by the dirt count values at the 0.05% and 1.0% levels of addition of NaCMC. Anionic surfactants do not appear to be compatible with NaCMC, precipitating the ink particles out as large globules that become

to large to float to the surface and during sheet formation re-deposit on the sheet surface. The brightness tests indicated that the addition of NaCMC, while decreasing ink speck count, does not measureably increase brightness. The brightness obtained from the handsheets at the 5.0% level of NaCMC was decidedly less than the value obtained for the peroxide treated handsheets. There was much more scatter in the whiteness data at the different levels of NaCMC addition; this phenomenon could indicate that whiteness is a more sensitive test for the effect of NaCMC on color. The whiteness test could be considered in future experimentation of being more indicative of what actually occurs. The tensile data collected was quite scattered and due to the non-uniformity of the handsheets is not significant. The sheet formation, due to the fact that no dilution water was added, causing the handsheet formation to be radical and sheet formation is critical, especially in tensile, since only a small area is being tested. In the study of pH vs NaCMC indication is that pH 10.0 is the optimum pH level yielding maximized value in dirt count, brightness and mullen with comparative values in tensile and whiteness.

Future Research

Based on the experimental analysis many questions are unanswered, especially in the area of maximizing the optimum level of NaCMC addition. If there is a detrimental NaCMC level of addition, what is the level or range. Produce sets of handsheets under ideal sheet

formation conditions with varying level of NaCMC addition and evaluate for strength. Determine the compatibility of NaCMC with peroxide formulations, especially in the area of dirt specks and strength. Determine why NaCMC observes incompatibility with anionic surfactants.

TABLE 1

NaCMC INDUSTRIAL VISCOSITY RANGES

VISCOSITY TYPE	DEGREE OF POLYMERIZATION	MOLECULAR WEIGHT
High	3,200	700,000
Medium	1,100	250,000
Low	400	90,000

TABLE II

CONSTRUCT OF ABSORPTION INKS

CONTENT	LETTERPRESS NEWS	LITHO OFFSET NEWS
Pigment %	9-15	10-18
Mineral Oil, %	80-90	20-50
H/C Resin, %	-	10-30
H/C Pitch, %	2-5	2-5
Mineral Seal Oil, %	-	20-30

TABLE III

CMC	ADDITIVES		PH	DIRT COUNT	BRIGHTNESS	WHITENESS	MULLEN	TENSILE
0	1.0% HOOH, 0.1% Tx-100		11	9.54/gm	51.4	59.2	15.8	4.41
0	.075% Tx100, .025% CF10		10	9.40/gm	50.3	58.0	14.7	3.53
0.05	"	"	10	10.4 /gm	50.4	58.8	15.1	4.28
1.0	"	"	10	6.87/gm	50.2	58.3	16.1	4.39
5.0	"	"	10	6.14/gm	49.2	57.8	17.3	3.51
5.0	"	"	9	7.87/gm	48.0	56.1	15.3	4.06
5.0	"	"	11	7.92/gm	49.4	57.2	16.7	3.20
0	0.25% Tx-114		10	8.64/gm	50.2	57.8	15.1	3.91
0.05	"	"	10	8.36/gm	51.3	58.0	16.8	3.56
1.0	"	"	10	9.81/gm	51.1	58.4	14.2	3.91
5.0	"	"	10	8.00/gm	49.7	57.3	15.8	3.74
0	0.25% Tn 101		10	5.63/gm	48.9	56.2	15.0	3.29
0.05	"	"	10	5.37/gm	49.6	57.7	15.9	3.87
1.0	"	"	10	6.96/gm	50.2	58.2	14.9	3.66
5.0	"	"	10	4.71/gm	50.8	58.8	16.5	4.04

CMC	ADDITIVES		PH	DIRT COUNT	BRIGHTNESS	WHITENESS	MULLEN	TENSILE
0	0.25% Steol CS-460		10	9.28/gm	49.9	57.8	14.6	2.73
0.05	"	"	10	7.18/gm	50.1	58.0	15.4	3.24
1.0	"	"	10	10.9 /gm	50.1	57.9	15.3	2.79
5.0	"	"	10	8.08/gm	49.5	57.2	18.1	3.1

NOTE: LARGE DIAMETER INK SPECKS (ANIONIC)

0	0.50% Wa-Extra		10	5.81/gm	49.3	56.8	13.7	3.11
0.05	"	"	10	11.0 /gm	47.9	56.9	14.3	2.76
1.0	"	"	10	7.52/gm	49.8	57.2	13.4	2.76
5.0	"	"	10	5.62/gm	49.7	57.4	16.0	3.06

0	0.25% Bio Soft LD-95		10	5.80/gm	48.5	57.3	14.5	2.92
0.05	"	"	10	6.02/gm	49.0	57.4	14.8	3.02
1.0	"	"	10	4.75/gm	49.2	57.0	15.3	2.83
5.0	"	"	10	5.02/gm	50.2	57.9	17.8	3.31

Figure 1

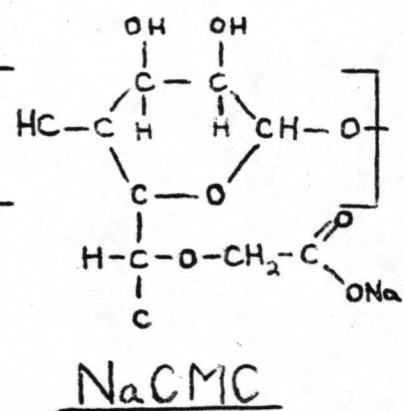
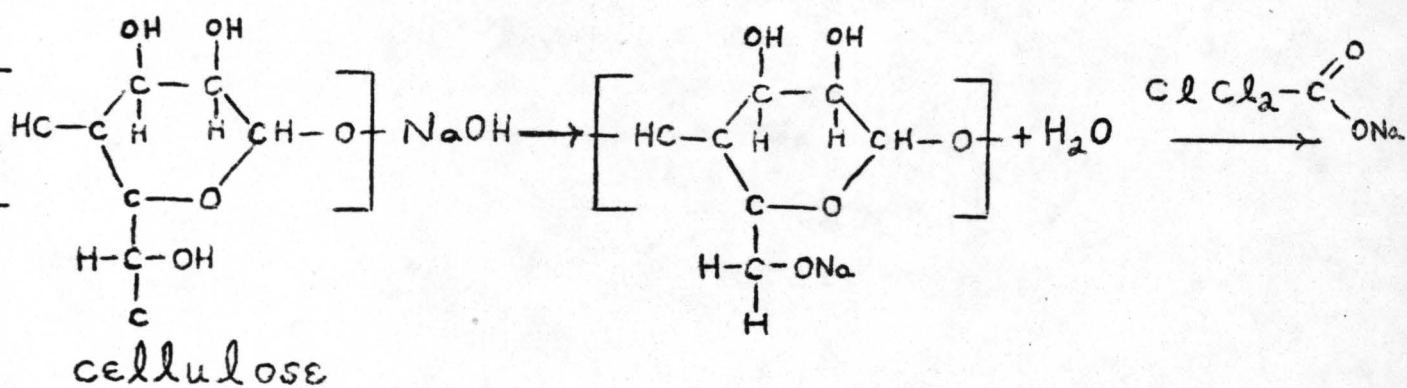
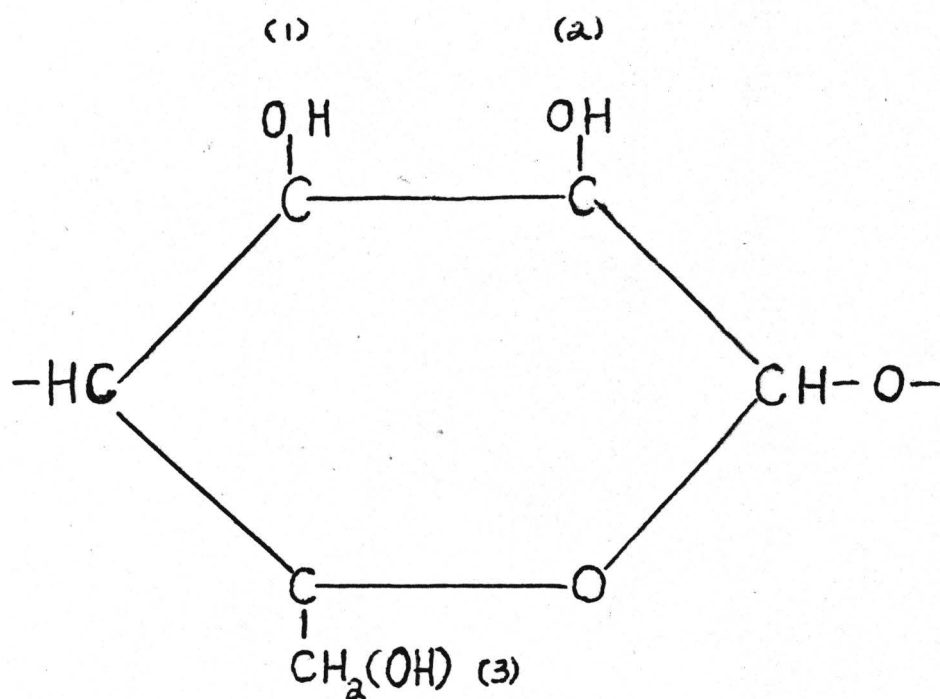


Figure 2



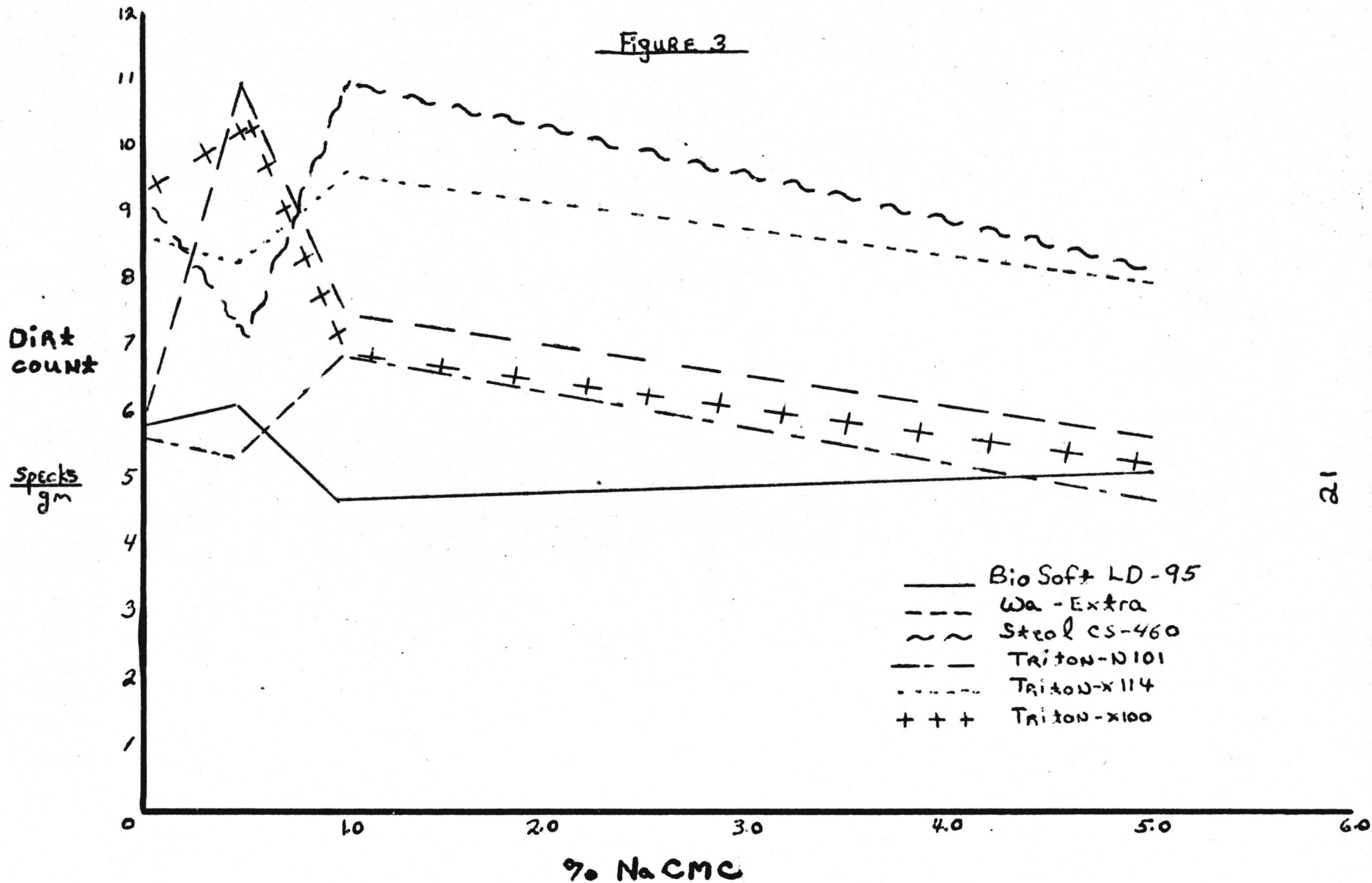


Figure 4

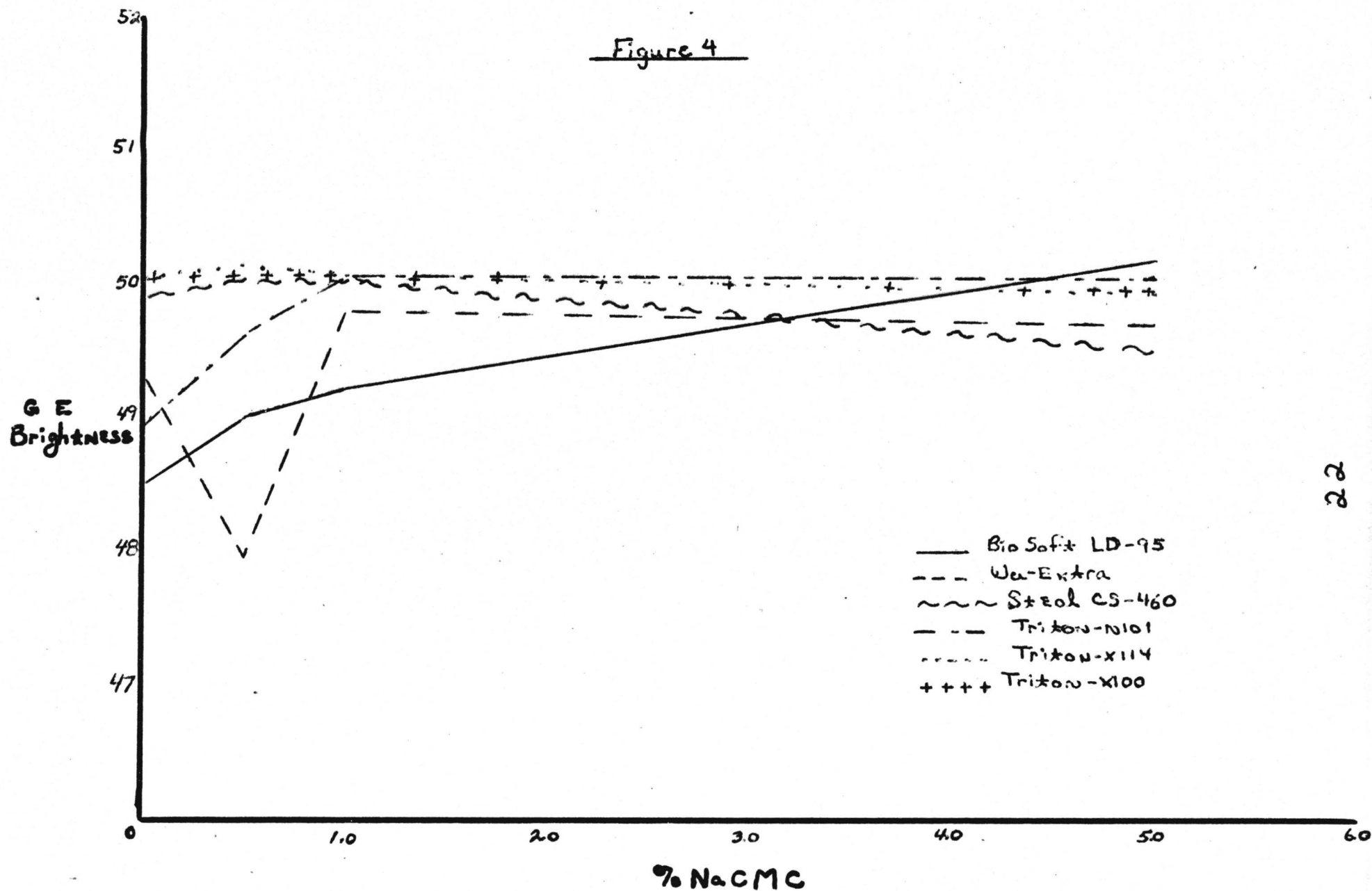


Figure 5

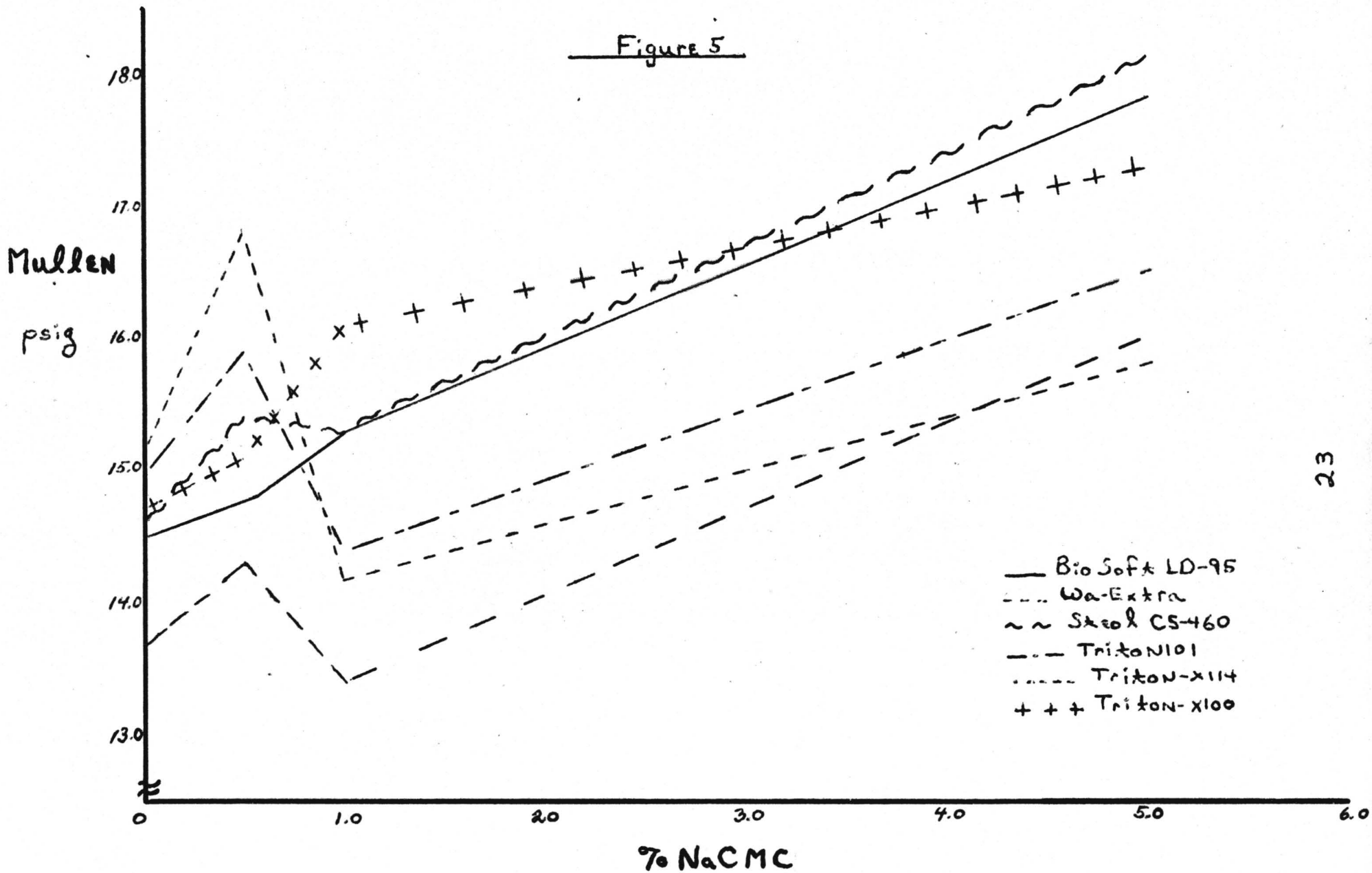


Figure 5

GE
Whiteness

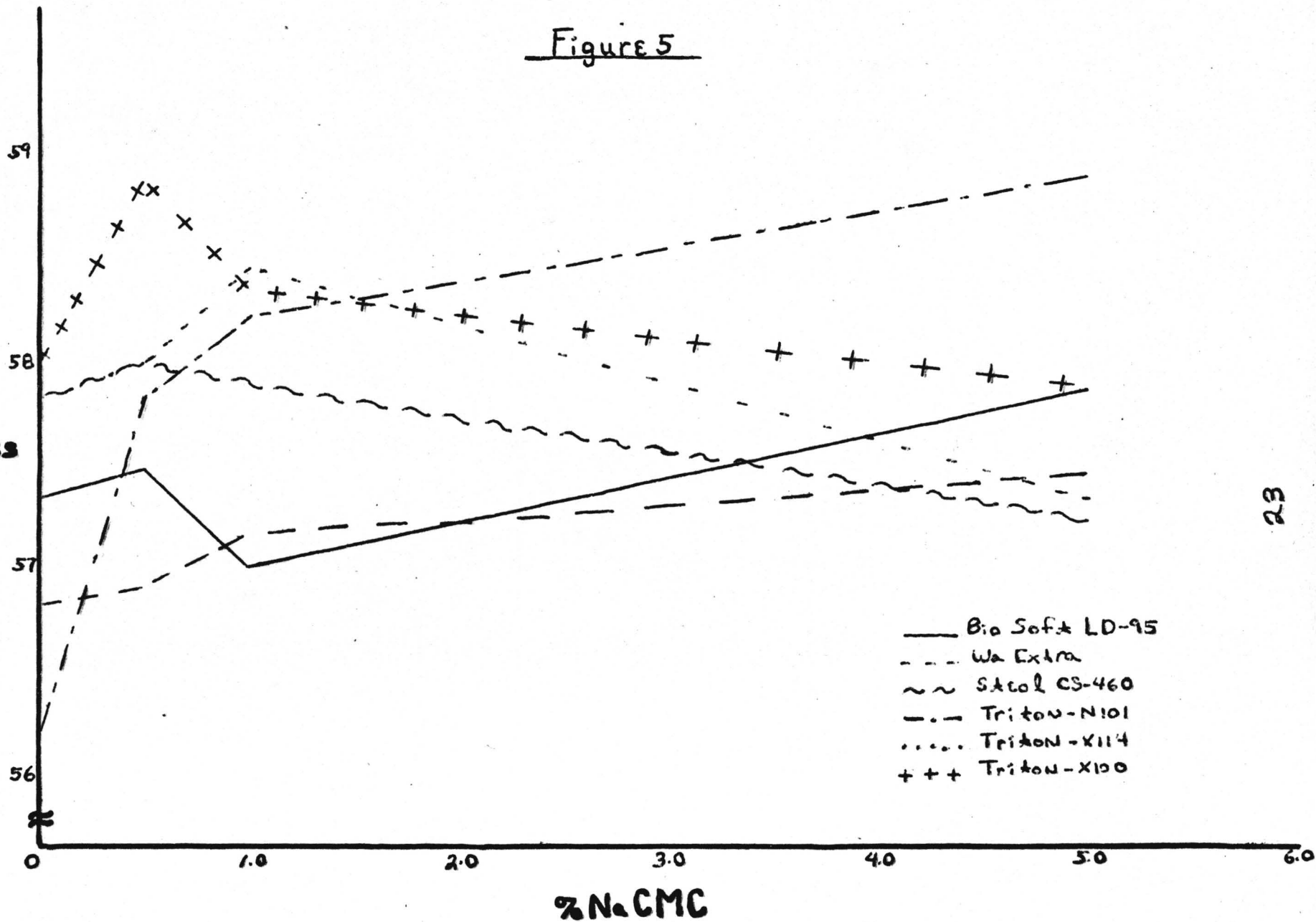


Figure 6

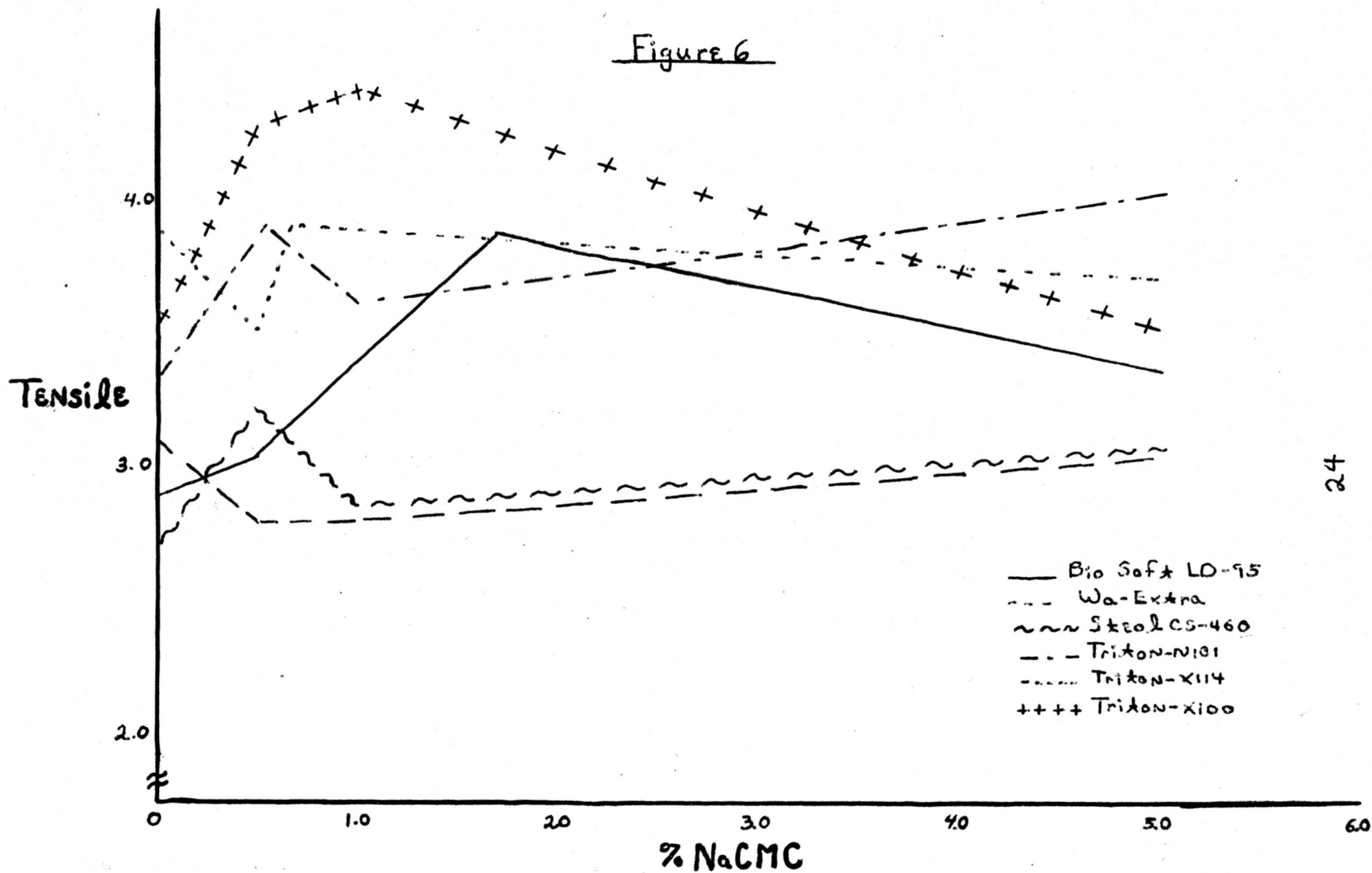


Figure 7

Na CMC ~ content remains
constant at the
5% level of ad-
dition

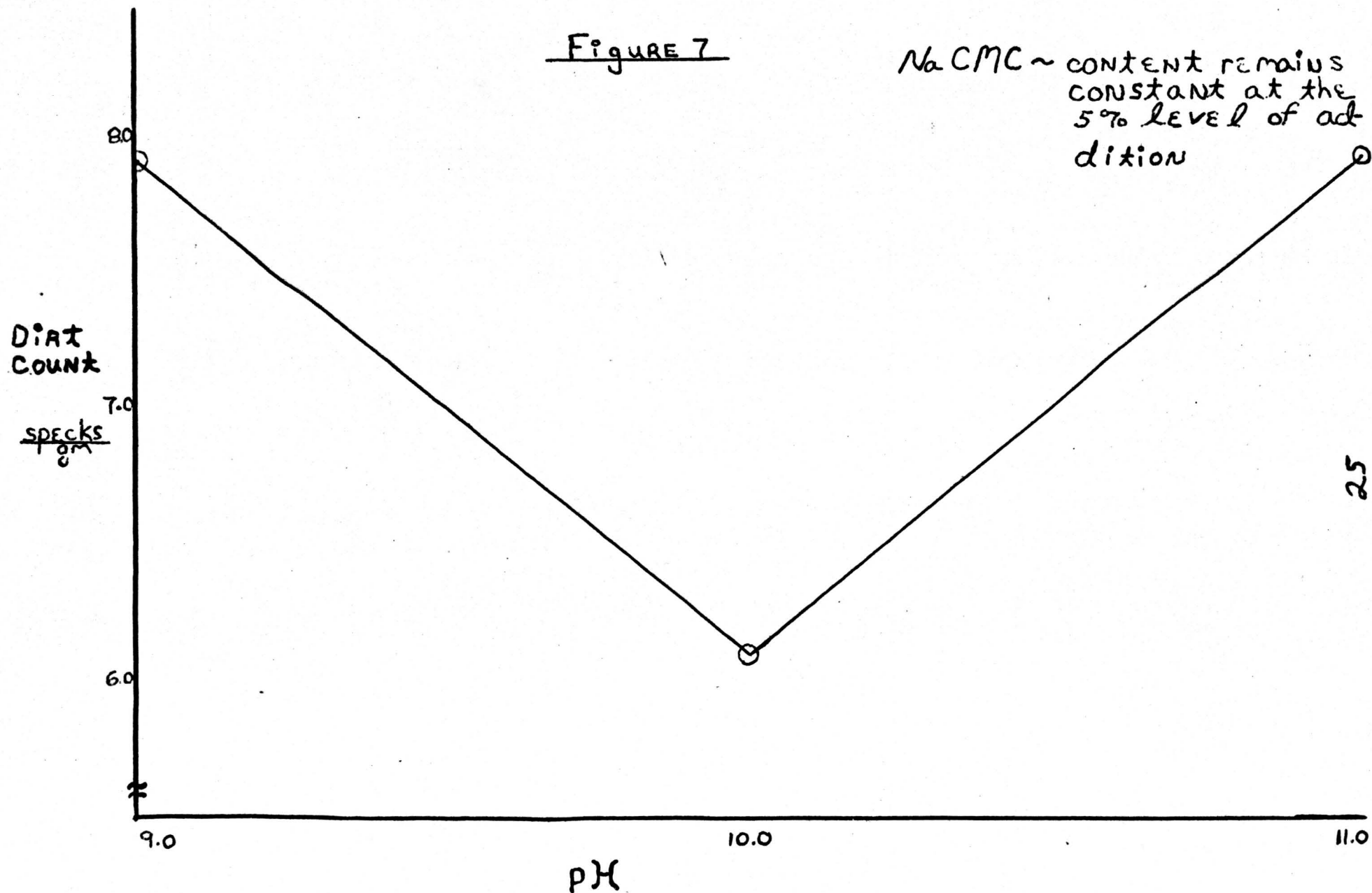


Figure 8

NaCMC content remains
constant at the
5% level of ad-
dition

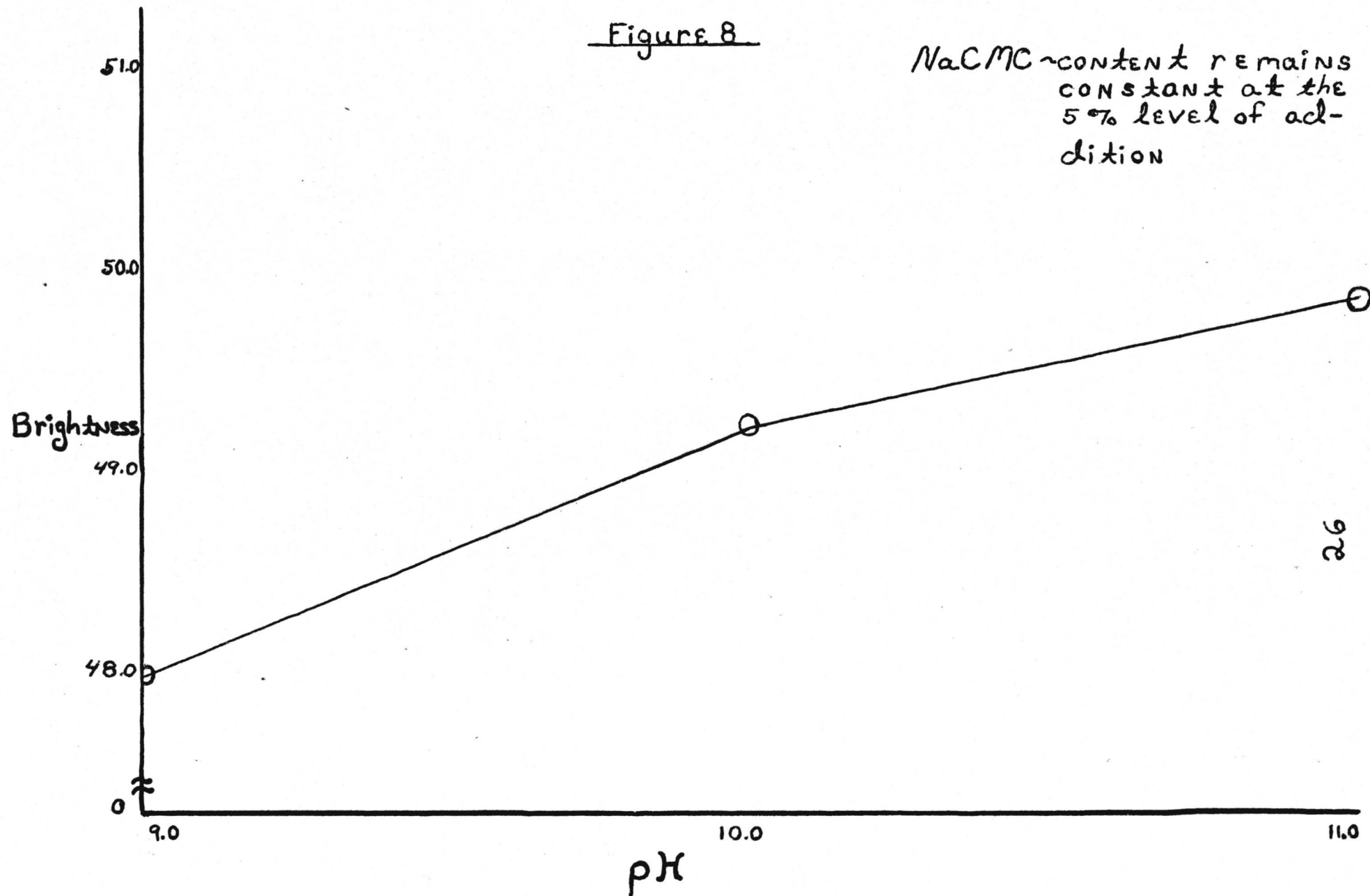
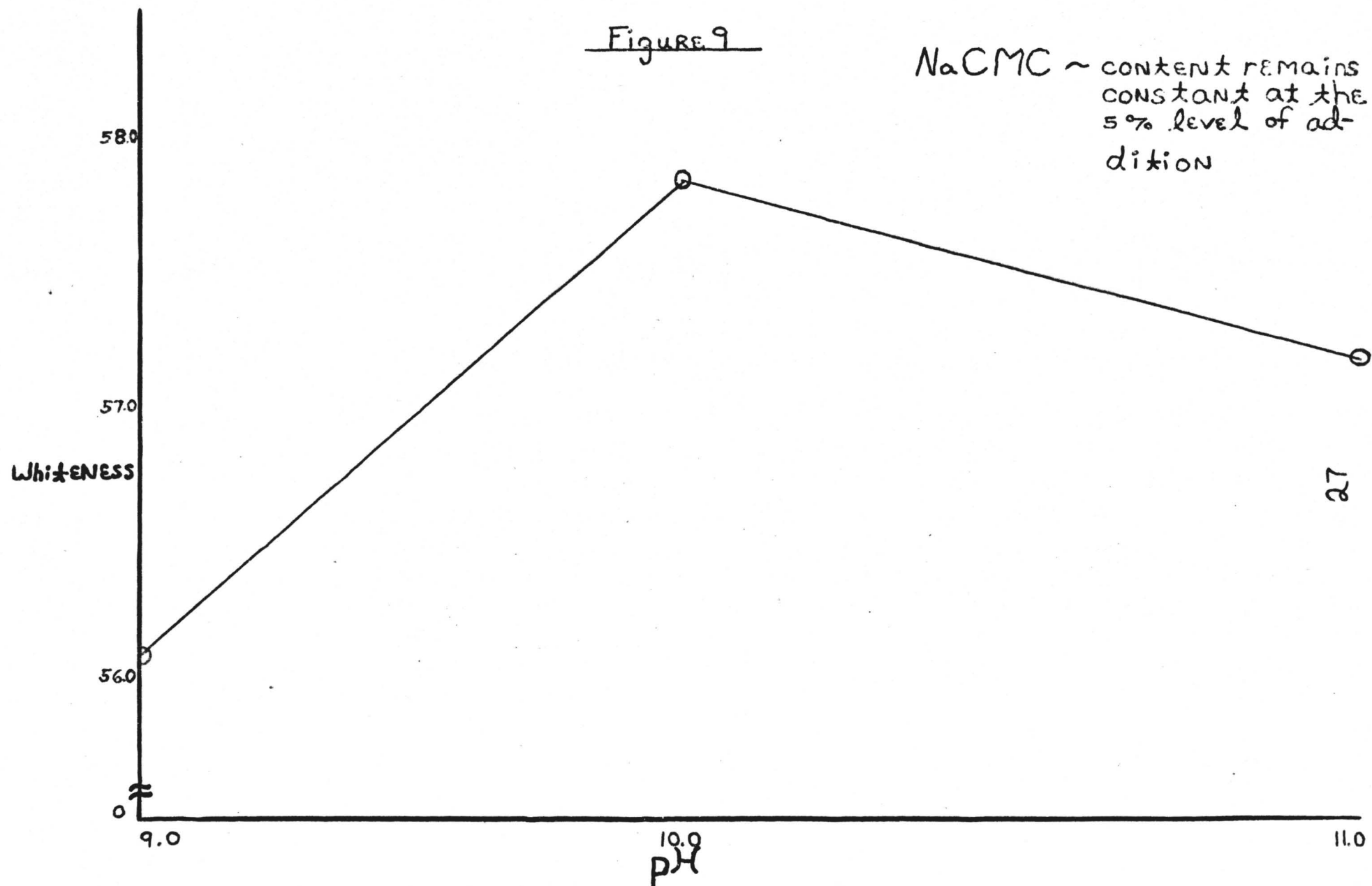


Figure 9

NaCMC ~ content remains
constant at the
5% level of ad-
dition



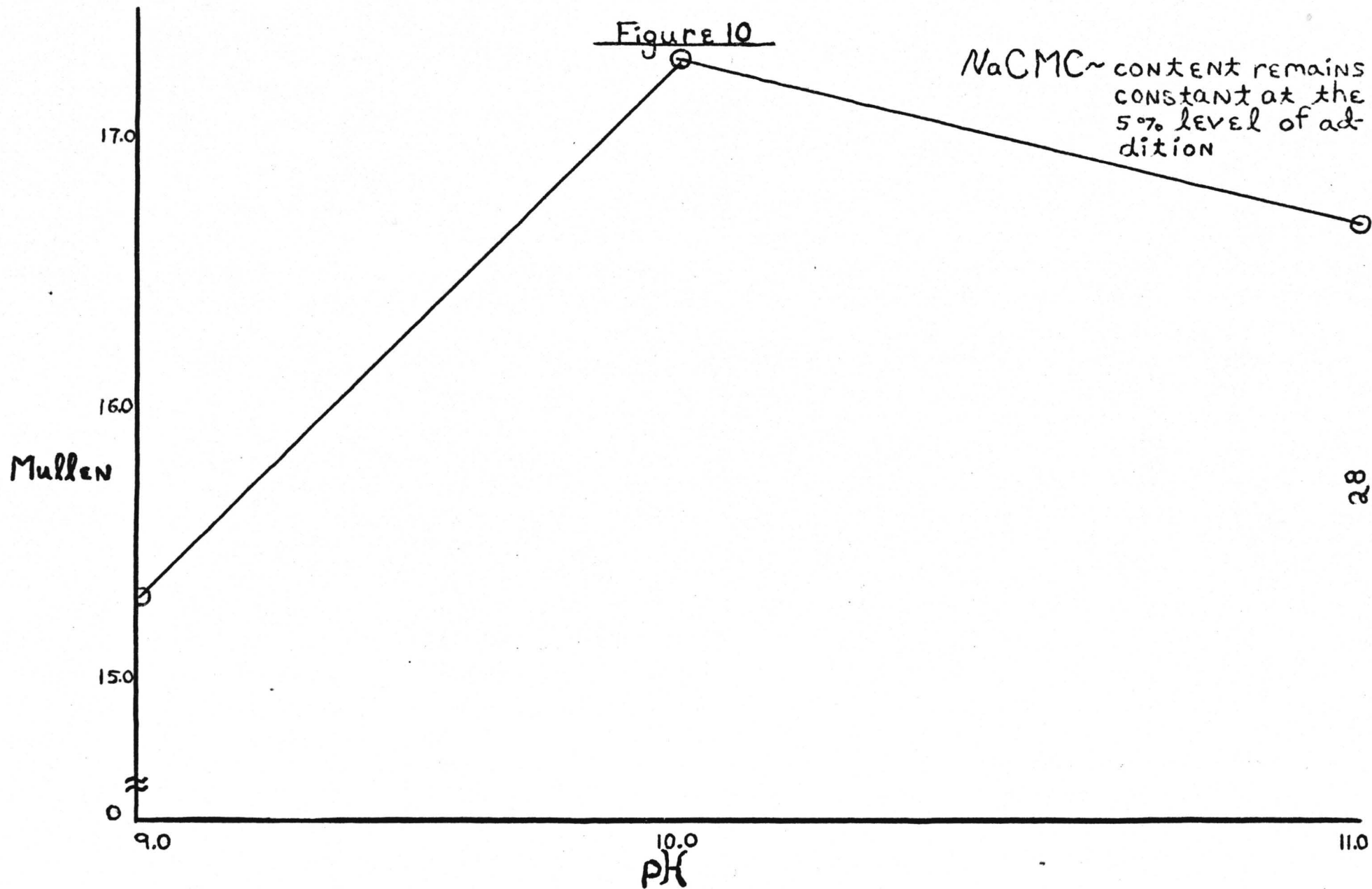
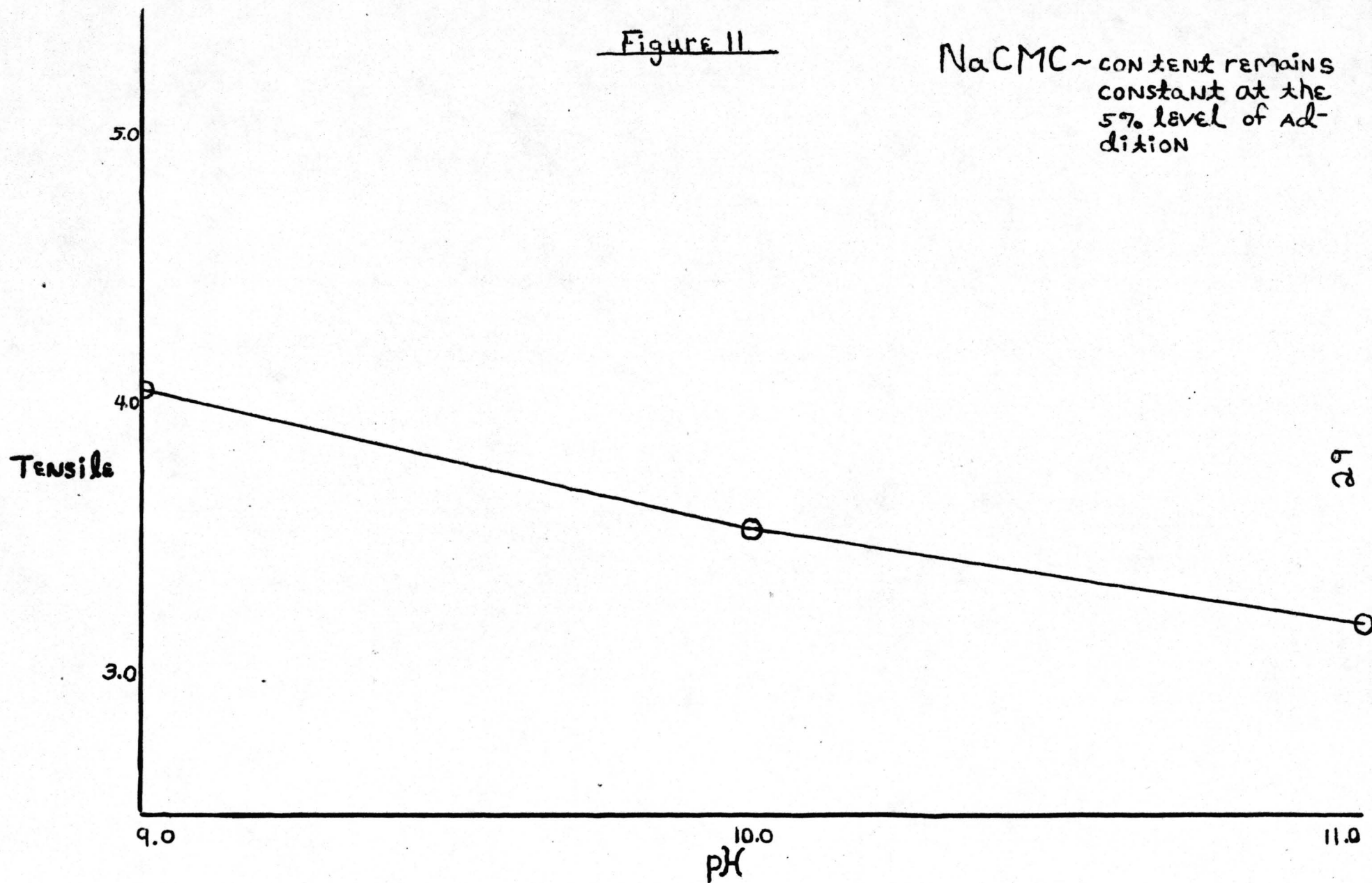


Figure 11

NaCMC ~ content remains
constant at the
5% level of ad-
dition



Footnotes

¹N. Pipel, The Use of NaCMC in Synthetic De-Detergents.

²Ibid.

³NaCMC in Liquid Detergents, J. B. Batdrof, Soap and Chemical Specialities

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⁵Pipel, op. cit.

⁶Pipel, op. cit.

⁷G. Bechstein, "Chemicals in the Drinking Process," Das Osterruchischs Papier, April, 1975, p. 74.

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