



4-1984

## The Effects of Different Carboxymethylcellulose Molecular Weight Grades with Polyamide Epichlorohydrin Resin on Wet and Dry Paper Strength

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THE EFFECTS OF DIFFERENT CARBOXYMETHYLCELLULOSE  
MOLECULAR WEIGHT GRADES WITH POLYAMIDE EPICHLOROHYDRIN RESIN  
ON WET AND DRY PAPER STRENGTH

by

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A thesis submitted  
in partial fulfillment for  
The Bachelor of Science Degree

Western Michigan University

Kalamazoo, Michigan

April, 1984

## ABSTRACT

Four different molecular weight grades of carboxymethylcellulose were used in combination with polyamide epichlorohydrin resin to study wet strength development. Noble and Wood handsheets were made with deionized water, using a white water recirculation system. Resin was added to thick stock followed by CMC, in dilute conditions. Wet and dry tensile strength were tested. Increasing CMC molecular weight increased dry strength. Increased CMC molecular weight had no effect on wet strength. CMC addition with resin increases wet strength.

CMC increases wet strength by a carboxyl group reaction with resin which results in increased resin retention. It was concluded that increased dry strength resulting from CMC addition, occurs by a bridging mechanism.

Keywords: Carboxymethyl cellulose; Polyamides; Wet Strength; Dry Strength.

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## Introduction

In the tissue and toweling industry the price of achieving wet strength with absorbancy and softness in paper has always been high. The internal addition of dual polymer systems have been very common in developing these properties at lower total chemical costs. One of these internal wet strength systems has been the combination of cationic polyamide epichlorohydrin resins with the sodium salt of carboxymethylcellulose. Many paper mills currently use this combination of internal additives. So far, no one has researched the mechanisms and wet strength effects of different molecular weight grades of carboxymethylcellulose when used in conjunction with polyamide epichlorohydrin resin. The major objective of this study is to examine the wet and dry strengths resulting from the use of different molecular weights of the carboxymethylcellulose in combination with this resin.

## THEORETICAL DISCUSSION

### Wet Strength Mechanisms.

It is known that paper in the dry state is held together by fiber to fiber bonds. These linkages are made up of hydrogen bonds between fibrils or amorphous cellulose regions which are pulled together by surface tension in the drying process. Paper which is untreated has relatively little wet strength because water penetrates the bonded areas. Once penetrated, this bonded area is effected by strong polar forces of the water breaking the

hydrogen bonds present in the dry state. Britt (1) showed that the strength loss of paper when wetted can be related to the wetting solvent's intensive solvation properties. In this study, paper was immersed in different solvents and the wet tensile strength was determined. The results showed that highly polar, low molecular weight solvents (like water), yielded the lowest wet strengths of paper.

Wet strength development by the use of resins can occur by five possible mechanisms (2). The first three alternative mechanisms are the formation of covalent, electrostatic, or valence bonds between the cellulose and the resin molecules. These bonds are much more resistant to water than are the hydrogen bonds due to their higher enthalpies of formation. The fourth mechanism theorizes that the fiber to fiber hydrogen bonds are surrounded by cured resins which prevent water from penetrating the bonded area. In the last theory, wet strength additive is diffused into the fiber surface and forms water resistant bonds by mechanical entrapment of the crosslinked resin. In all these cases the wet strength development is usually also dependent on the curing of the resin.

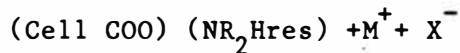
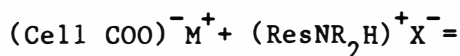
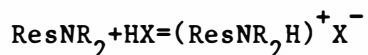
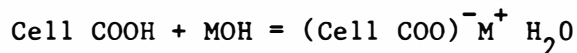
These separate mechanisms usually apply to each separate resin configuration in a different manner. Glyoxyl resins (2) are believed to create wet strength by forming covalent bonds with the fibers, however no concrete evidence is available to prove it. There are also many clues that show that Melamine formaldehyde resins (3) may also create covalent bonds in paper. Polyethyethylenimine resins are thought to create bridging by their long polymer chains along with some type of sorbtion or bonding with fibers to create wet strength.

Polyamide epichlorohydrin resins will be used in this dual polymer system study, therefore it is important that their wet strength development mechanism is understood. Bates (2) found that the creation of covalent bonds from the use of polyamide epichlorohydrin resins were not a large factor in the wet strength development process. He also found that the cured resin helped create wet strength by keeping the water away from the hydrogen bonds formed in the paper. However, he did find that the cured resin did not completely exclude water from the fiber to fiber bonded areas. Bates concluded that only 10% of polyamide epichlorohydrin monomer units had reactive sites which were involved in the curing reaction which led to the water proofing of the cellulose bonds. Another important wet strength mechanism which could be involved is the intermixing of the resin with the fiber polysaccharides in the resin retention and diffusion stages at the wet end. When the paper is dried and the resin cures, the polysaccharide provides a mechanical entrapment for the crosslinked resin. There is no actual proof that these wet strength mechanisms are correct with the use of polyamide epichlorohydrin resins, however indirect evidence points towards these theories.

#### Wet Strength Resin Retention

The retention of wet strength resins are very important to efficient wet strength development. Both polyamide epichlorohydrin and polyethylenimine resins are thought to be retained by carboxyl groups on the pulp by an ion exchange mechanism (4,5,6). The ion exchange resin retention mechanism works

as follows:



M= metal, Res= Wet strength resin

Bates (4) proved this mechanism by qualitatively showing one equivalent of calcium ions on the pulp is lost for every 2 repeating units of the resin that was retained. From this study he concluded that the polyamide epichlorohydrin resin polymer is adsorbed tightly to the fiber surface and does not contain long ends of the polymer chain hanging off into the solution. The resin is not easily desorbed by shear forces after it has been adsorbed by the fiber.

The speed of the retention of polyamide epichlorohydrin resin into a .02% consistency stock was shown to be very fast and reached a constant retention level within minutes. The retention of polyamide resin is never a problem if there are carboxyl groups present on the pulp along with some metal ions in solution to permit the ion exchange mechanism to occur. It must also be noted that the presence of too many ions in low consistency stocks will reverse the ion exchange reaction and force the resin to desorb off the fiber.

Bates (4) also showed that increasing the surface area of the cellulosic material had no effect on polyamide epichlorohydrin resin demand. This was demonstrated by low retention of resin on Avicel (a pure microcrystalline cellulose with high surface area and small amounts of carboxyl groups).

However, when the Avicel was oxidized to form carboxyl groups, retention of the resin increased in proportion with the increase in carboxyl groups which, once again, demonstrates the ion exchange retention mechanism.

Chiu (7) Studied the use of the Nobel and Wood handsheet former and the amount of resin build up in the white water system using .5% polyamide epichlorohydrin resin addition. Following 9 recycles of white water the resin retention is very high in this dilute handsheet process.

Another important part of wet strength resin retention is the amount of resin that stays on the surface of the fiber as opposed to the amount of resin which diffuses into the fiber (8). Strazdins (9) showed that when polyamide epichlorohydrin resin was added to oxidized pulp the resin diffused deeply into the fiber requiring high amounts of polymer to neutralize the fiber surface charge density. In opposition to this, Becher (10) concluded that Melamine formaldehyde resin does not diffuse into the fiber appreciably at papermaking concentrations. Since there are differing retention mechanisms of these two resins it can be concluded that the exact amount of polyamide epichlorohydrin resins that diffuses into the fiber is not actually known, however it may relate to the amount of carboxyl groups on the internal surfaces of the fiber.

#### Polyamide Epichlorohydrin Resins

Polyamide epichlorohydrin resins are important in the manufacture of absorbant wet strength papers. This cationic resin is more expensive than the others but the extra additive cost yields paper with permanent wet strength in combination with a soft feel and high absorbancy. Polyamide epichlorohydrin

cationic resins have the benefit of being PH insensitive unlike Melamine formaldehyde resin where increased or decreased PH cause differences in charge density resulting in changes in resin retention.

Polyamide epichlorohydrin resins are manufactured by a process similar to that used to produce Nylon 66 (11). The major difference is that the resin contains secondary amine groups which makes the polymer soluble and creates sites for the addition of epichlorohydrin groups. The resin is produced by reacting a condensation polymer from adipic acid and diethylenetriamine with epichlorohydrin. The polymer is given its cationic charge by the epichlorohydrin alkylation converting the secondary amine to a quaternary nitrogen. Also present along the polymer chain are primary and secondary amine groups.

#### Carboxymethylcellulose.

Carboxymethylcellulose, or CMC, is an chemical derivative of pure cellulose. CMC is produced from the reaction of monochloroacetic acid with cellulose in a solution of strong alkali. The CMC is an extremely hydrophilic material and contains a cellulose backbone chain with various units on the chain containing carboxymethyl groups. The amount of these carboxymethyl groups per monomer unit is known as the degree of substitution. These water soluble anionic polymers are manufactured at various degrees of substitution and molecular weights. Degrees of substitution range from .4 to 1.2 and molecular weights are available from 90,000 to 700,000.

The main use in the past for internal addition of CMC was for the development of dry paper strength (12). The mechanism of dry strength development is as follows. The CMC is adsorbed onto the fiber and upon drying, the capillary action of the water pulls the CMC into the fiber to fiber bonded areas. In these bonded areas the CMC could bridge the two fibers producing extra bonds, therefore yielding higher paper strength. The CMC creates no additional wet strength due too the fact that the CMC to fiber bond is easily dissociated by water. CMC retention in papermaking systems is low and therefore limited in its strength development.

#### Polyamide Epichlorohydrin Resin and CMC Systems.

It has been found that the combination of CMC with polyamide epichlorohydrin resins show dramatic increases in wet strength that are not present by the resin alone. Since CMC does not promote wet strength when used as a dry strength additive, the CMC must somehow react with the resin for this extra wet strength development.

Early work done by Raff (13) showed that surface application of CMC and polyamide epichlorohydrin resin improved the strength of paper after artificial aging. Another study performed by Neogi (14) showed the effect of the internal use of CMC with a sprayed addition of polyethyleneimine. In his results he attributes a large increase in wet strength to the anionic CMC adding negative charges to the system therefore creating ionic bonds between the CMC and resin. Another related wet strength system is an epoxylpropyl starch modified with epichlorohydrin groups which produced

a large increase in wet strength (15). The results of this study led to the conclusion that the epoxylpropyl group was the reactive site for the covalent bonding of the starch and cellulose.

The latest work by Espy (16) extensively studied the dual polymer wet strength system of carboxymethylcellulose and polyamide epichlorohydrin resin. This study showed the factors involved in optimizing chemical furnish and outside factors which must be considered in different wet end systems. The addition of resin without CMC increases dry strength and wet strength. Increasing additions of CMC while keeping resin furnish constant increases both wet and dry strength up to a maximum level where extra CMC lowers both strengths. The optimized CMC to resin furnish is based on the mole ratio of CMC carboxyl groups to each formula weight of resin. Wet strength is optimized at a weight ratio of .46 CMC (D.S=.7) to polyamide resin, which also relates to the isoelectric point of the resin-CMC mixture. The optimum ratio was based on deionized water and can be higher for differing wet end conditions. He concluded that over usage of CMC tended to deplete the resin from the fiber resulting in lower strength. It was also found that hard water systems were less prone to overusage of CMC and had optimum strength development over a wide range of weight ratios. Also studied was the importance of order in the addition of the CMC and resin to the pulp. It was concluded that the addition order of the polymer was insignificant to strength development if CMC addition was not too high. If CMC furnish is beyond the optimum level before resin is added, the CMC will compete with the fiber in the resin retention stage. In this

situation, mobile CMC molecules will surround the resin polymer neutralizing its charge thus reducing its attraction to fiber, causing poor retention and bonding which results in lower wet strength.

Overall many possible wet strength development mechanisms of the CMC, polyamide epichlorohydrin resin systems must be considered. One possible mechanism is from a charge distribution point of view. Cationic charges of polyamide epichlorohydrin resins are neutralized by CMC resulting in a lower positive charge on the fibers. This lower charge brings the system closer to the isoelectric point and increases resin retention causing higher wet strength. Another possibility is the CMC has negative charges which in solution could help decrease the diffusion of the cationic resin into the fiber. With the resin on the surface of the fiber it can be more efficient in bonding to develop wet strength and move chemical on the surface during the curing reaction. Direct bond of the resin and CMC via the proven ion exchange mechanism as explained earlier, is probably the best explanation for increased wet strength with the use of CMC. The CMC chains would be able to bridge between two sites of retained resin and create a bond which is resistant to water. A capillary action drawing the CMC into the contact areas of the fiber upon drying could be involved in this resin to CMC bond (17).

#### PRELIMINARY WORK

To determine the effect of white water recirculation and the use of tap water in the handsheet process, preliminary papermaking and paper testing runs were made at various conditions. The use of tap water with resin led to severe and inconsistent brightness losses in paper made in the handsheet system. Large amounts of metal ions interacting with resin components can

account for this phenomena. This problem did not occur when deionized water was used with resin or tap water was used without resin. The yellowing inconsistencies may be due to the irregular ion concentration in the water supply. From these observations it was concluded that deionized water would eliminate ion concentration variability and should be used for this study.

It was decided that white water recirculation would be used in order to simulate wet end conditions and reduce deionized water consumption. The white water recirculation gave slower drainage than the normal drain valve, however, this did not present a significant problem.

## STATEMENT OF PROBLEM

The use of polyamide epichlorohydrin resins is very common in the paper industry for wet strength development in paper. This resin offers many runnability and end use advantages in paper, however, it is very expensive. From previous work, it was shown that CMC used with polyamide epichlorohydrin resin enhances wet strength development. In this system, inexpensive CMC addition can reduce resin furnish while yielding the same wet strength at a lower cost. Presently, medium molecular weight CMC grades (Molecular weight of 250,000) are used in these applications. To this point no study has documented the effects of using higher or lower molecular weight grades in wet strength development. The objective of this thesis is to study the different effects of CMC molecular weight grades when used in combination with polyamide resins. The secondary objective is to make observations and suggestions for wet end additive studies on the Noble and Wood handsheet former.

## EXPERIMENTAL DESIGN

Handsheets were made on the Noble and Wood handsheet former with deionized water using the white water recirculation system. At the start of each run, 4 handsheets (not tested for strength) were made with deionized water to fill the recirculation system.

One per cent resin was added to a master pulp batch. Predispersed CMC was added to the proportionator at a level of .4%. Handsheets were made at five different conditions which included resin without CMC and each of the 4 different molecular weight grades available.

To eliminate variability, large sample sizes were tested for wet and dry tensile strength. To eliminate weight variation, the adjusted strength values were used in the final data evaluation.

## EXPERIMENTAL PROCEDURE

### Materials

#### Pulp

Throughout the duration of this experiment northern hardwood and softwood bleached kraft pulp in dry lap form were used. Pulps were blended 50:50 by weight and were refined in a Valley beater using deionized water. Refining was performed in accordance with TAPPI standard T-200 OS-70 to a Canadian Standard Freeness of 550 cc.

#### Polyamide Epichlorohydrin Resin

Polyamide resin is commercially available from Hercules Inc. under the product name of Kymene 557 H. Resin was stored at 5 degrees C to minimize loss of efficiency over an extended period of time. It has been shown that this resin has a high affinity for glass surfaces, therefore plastic containers were used for all treatments.

#### Carboxymethylcellulose

CMC was also available from Hercules Inc. Grades with a .7 degree of substitution were selected for this study. The four different molecular weight grades used (listed in increasing molecular weight) were: CMC 7LT, CMC 7MT, CMC 7M31T, and CMC 7M65T.

## Water

Water was deionized using a Culligan deionizing system. This system consisted of a water softener tank followed by two resin bed deionizer tanks. Tap water was not used in any pulp or paper processing.

## Noble and Wood Handsheet Former

The Noble and Wood handsheet former was used to produce standard handsheets using a white water recirculation pump for dilution water.

## Handsheet Procedure

The standard hardwood-softwood stock (approximately 2.0 consistency) was mixed in a bucket with a large laboratory agitator. The polyamide epichlorohydrin resin was added to this master batch of pulp at 1% dry resin to dry pulp basis. The resin was diluted with deionized water (for good dispersion) and added to the pulp under vigorous agitation. The pulp was then agitated mildly for 5 minutes before samples were taken to fill the proportionator. Samples of this pulp were added to the proportionator and a sheet was made with a liter sample. At this time the CMC was weighed and predispersed in deionized water with a high shear mixer. The proportionator was diluted to make 2.5 gram handsheets by adding the predispersed CMC and water. Four sheets were made using the recirculation drain valve which filled the recirculation tank with white water. These four sheets were then discarded. The test sheets were made using the dilution valve to fill the mold with recirculated white water and a stock sample from the proportionator. The mold was drained back to the

recirculation tank and the sheet was pressed and then dried at 240 degrees F. Sheets were produced until the proportionator was empty. This handsheet making procedure was repeated for the four separate CMC grades.

#### Handsheet Curing

After the sheets were prepared, they were heat cured in a forced air oven at 105 degrees C for 1 hour. After curing, the sheets were put in a standard conditioning room for 24 hours.

#### Handsheet Strength Testing

To test the dry strength of the handsheets, an Instron tensile tester was used. Samples were cut with a double edged paper cutter to a 15mm width. Samples were tested in the Instron at a rate of elongation equal to 2cm per minute and a sample jaw separation of 10cm. Approximately 30 wet and dry samples were tested for each treatment.

For wet strength testing, the cut paper samples were laid on a coarse mesh screen and covered with another screen. They were then submerged under 3 inches of deionized water for 5 minutes. The samples were removed from the water and the top screen was removed. Each sample was slid onto a blotter paper to remove excess water. The wet paper strips were then tested for tensile in the same manner as the dry strips.

## RESULTS

### Wet Strength

Wet strength data did not show any consistent correlation with increasing CMC molecular weight. When multiple T-tests were computed (see Table 2), certain CMC grades showed significantly higher wet strength, but this difference may be due to sampling error.

It can be concluded that different CMC molecular weight grades in combination with polyamide resin had no significant effect on wet strength (see Graph 1 and Table 1).

### Dry Strength

Data for dry strength comparisons were also evaluated with multiple T-tests (see Table 3). There were significant differences in the data along with trends for the dry strength development.

Increased CMC molecular weight (see Graph 2) improved the dry strength of paper. CMC65MT (the highest molecular weight grade) achieved the highest dry tensile strength. The incremental dry strength increases resulting from molecular weight changes were not significantly different in pair-wise comparisons at the .05 level. There is a trend of increasing dry strength with increasing molecular weight and various molecular weight comparisons are significantly different.

## DISCUSSION OF RESULTS

### Wet Strength

The absence of wet strength development with increased CMC molecular weight could be explained by several mechanisms. Higher molecular weight grades of CMC were found to promote bridging but not increase wet strength. It was concluded that increased wet strength gained with CMC addition to a resin

system does not occur by the CMC polymer bridging retained resin on adjacent pulp fibers. CMC is involved in the wet strength development. There may be a reaction between the carboxyl groups of CMC and the polyamide resin. This reaction could be involved in promoting higher resin retention resulting in higher wet strength. Charge interactions of the two polymers can also explain these results. The anionic polymer could help bring up resin absorbed in fiber surfaces giving better resin mobility in the crosslinking reaction, yielding more waterproof bonds.

#### Dry Strength

Dry strength improvements with increased CMC molecular weight could be explained by fiber to fiber bridging of the CMC polymer. With increased molecular weight the CMC can bridge more effectively between gaps of unbonded surfaces and create extra linkages making fiber to fiber bonds stronger. These dry strength bonds are water sensitive and have nothing to do with wet strength development.

#### CONCLUSIONS

- 1) Increasing CMC molecular weight when added with polyamide resin has no significant effect on wet strength.
- 2) Increased wet strength resulting from the addition of CMC to a resin system does not occur from CMC bridging. These wet strength increases are a result of CMC carboxyl groups and resin interacting possibly resulting in higher resin retention.

3) Dry strength improves with increased molecular weight grades of CMC.

This is due to increased fiber to fiber bonding resulting from CMC bridging.

#### RECOMMENDATIONS FOR FUTURE WORK

1) Find the effect of shear in this dual polymer system.

2) Examine if CMC helps retain cationic alkylketene dimer size and determine its effect on sizing.

## APPENDIX

Table 1

CHEMICAL ADDITION	NUMBER OF TEST SAMPLES	DRY TENSILE (KN/M) CORRECTED	WET TENSILE (KN/M) CORRECTED
NO CHEMICALS (CONTROL)	27	3.12	.15
1% RESIN NO CMC	30	3.92	1.10
1% RESIN .4% CMCLT	33	4.35	1.32
1% RESIN .4% CMCMT	33	4.56	1.39
1% RESIN .4% CMCM31T	36	4.64	1.31
1% RESIN .4% CMCM65T	30	4.73	1.35

ALL VALUES CORRECTED TO A 2.6g HANDSHEET WEIGHT

TENSILE Kg/15mm CORRECTED = UNCORRECTED TENSILE \* 2.6g/HANDSHEET WEIGHT GRAMS

TENSILE KN/M CORRECTED = TENSILE Kg/15mm \* 1.53

Table 2  
A Point By Point Statistical Comparison  
Between Wet Strength Sample Means

Chemical Treatment	No Chemicals	1% Resin No CMC	1% Resin CMCLT	1% Resin CMC MT	1% Resin CMC M3IT	1% Resin CMC M65T
No Chemicals						
1% Resin No CMC	← Significant Difference					
1% Resin .4% CMCLT	← Significant Difference	← Significant Difference				
1% Resin .4% CMCMT	← Significant Difference	← Significant Difference	← Significant Difference			
1% Resin .4% CMC M3IT	← Significant Difference	← Significant Difference	Not Significant	↑ Significant Difference		
1% Resin .4% CMC M65T	← Significant Difference	← Significant Difference	← Significant Difference	↑ Significant Difference	Not Significant	

95% Confidence Limit

→  
arrow points towards  
highest mean

$$H_0: \mu_1 = \mu_2$$

$$H_1: \mu_1 \neq \mu_2$$

$$t_{\text{critical}} = 2.000$$

if  $t > 2.00$  reject the Null

# Table 3

A Point By Point Statistical Comparison  
Between Dry Strength Sample Means

Chemical Treatment	No Chemicals	1% Resin No CMC	1% Resin CMCLT	1% Resin CMC MT	1% Resin CMC M3IT	1% Resin CMC M65T
No Chemicals						
1% Resin No CMC	← Significant					
1% Resin .4% CMCLT	← Significant	← Significant				
1% Resin .4% CMCMT	← Significant	← Significant	Not Significant			
1% Resin .4% CMC M3IT	← Significant	← Significant	← Significant	Not Significant		
1% Resin .4% CMC M65T	← Significant	← Significant	← Significant	← Significant	Not Significant	

95% Confidence Limit

→  
arrow points towards  
high mean

$$H_0 : \mu_1 = \mu_2$$

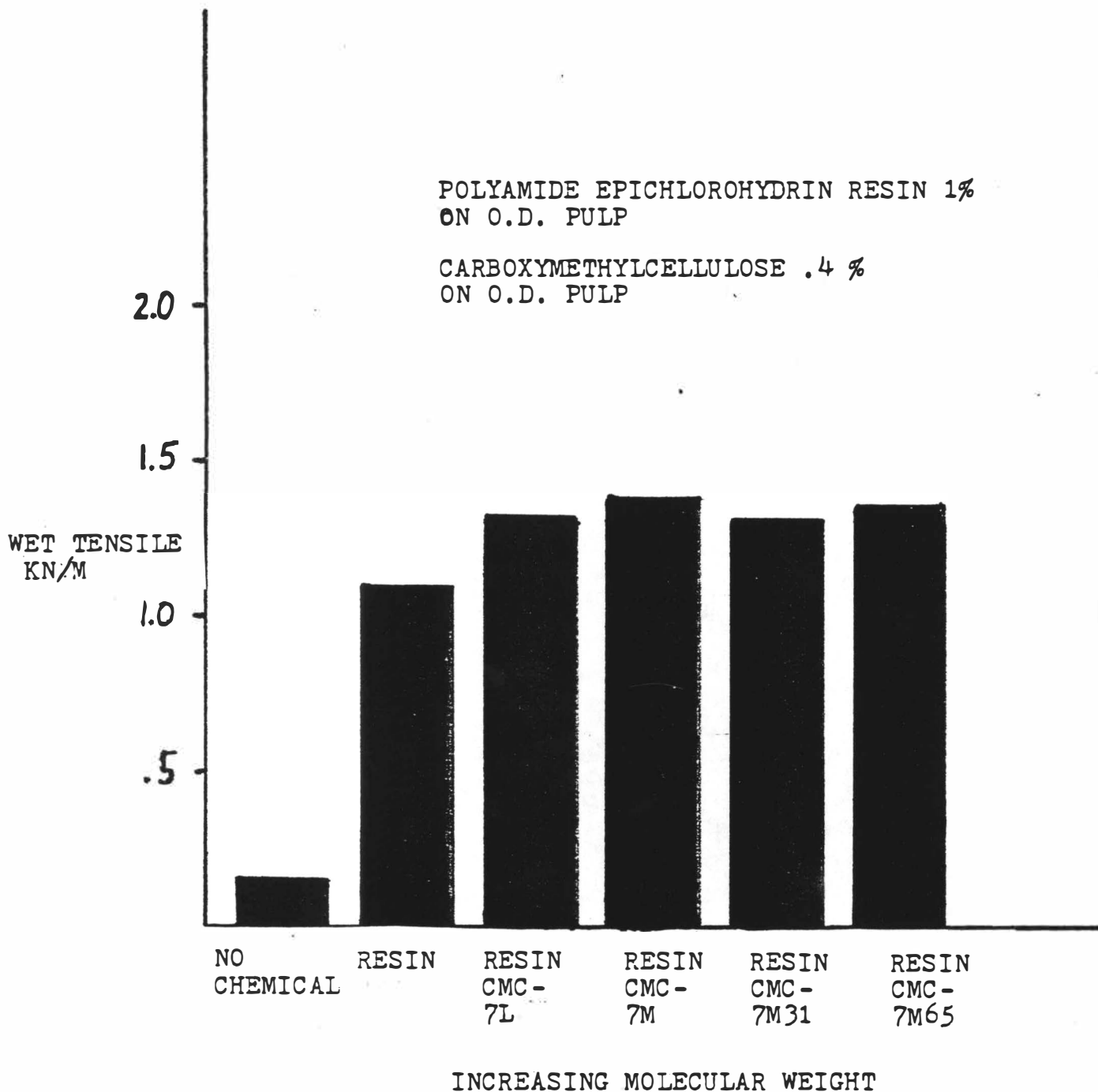
$$H_1 : \mu_1 \neq \mu_2$$

$$t\text{-critical} = 2.000$$

if  $t > 2.00$  reject the

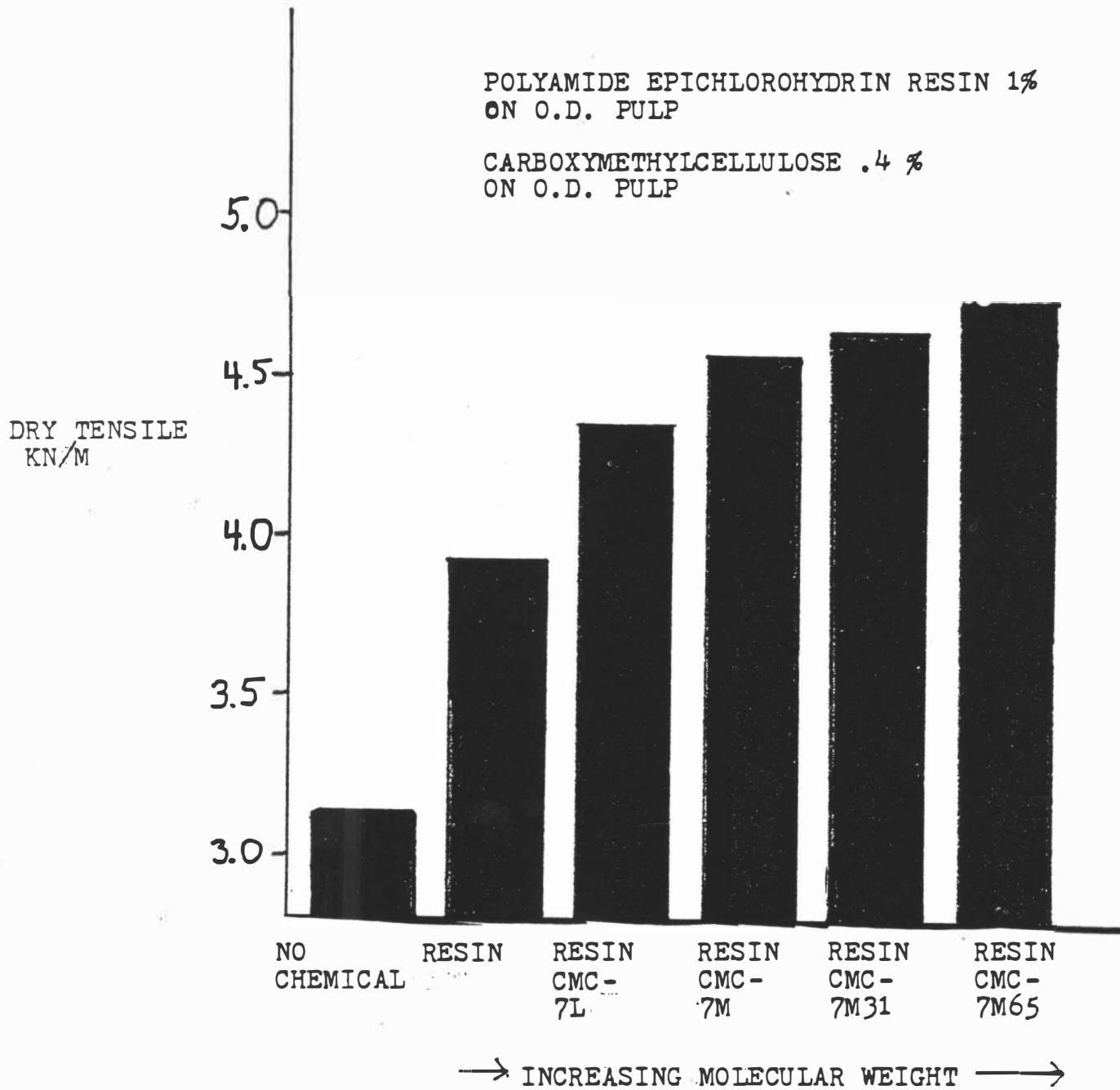
GRAPH 1

WET STRENGTH VS CMC MOLECULAR WEIGHT



GRAPH 2

DRY STRENGTH VS CMC MOLECULAR WEIGHT



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