4-1994

The Effects of pH and Soaking Time on the Repulping of Old Corrugated Containers (OCC)

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by

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A thesis submitted in partial fulfillment of the requirements for the degree of

Bachelor of Science

WESTERN MICHIGAN UNIVERSITY

Kalamazoo, Michigan

April 15, 1994

Western Michigan University

Abstract

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Efforts to increase production capacity and meet recycled fiber content regulations include the utilization of recycled OCC as a fiber source. This fiber source is predominantly softwoods which are characteristically longer and stronger than hardwood fibers. However, the corrugating medium found in OCC contains mostly hardwood fibers which are shorter and more brittle than the desired softwoods. In addition, the medium contains a significant amount of lignin that interferes with fiber bonding. Therefore, the resulting paper web's strength is decreased.

The paper industry is forced to find ways to achieve maximum strength from an inferior starting material. One method under investigation is caustic soaking. This study investigates the effects of pH and soaking time on the recycling of OCC and attempts to identify the optimum repulping conditions.

The OCC furnish was repulped at several pH levels. At each pH level, the stock was then soaked for various lengths of time. Subsequent testing of the pulps and handsheets, and analyses of the results were completed in order to determine the above mentioned objectives.

The effects of pH and soaking time on the OCC pulp and resulting handsheets are discussed in this report. The optimum repulping conditions were determined to be at a pH of 9 and a soaking time of 5 to 10 hours. Statistically significant strength increases in the handsheets produced at these optimum conditions were found to range between 5 and 19 percent for the various tests performed.

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Chapter 1: Introduction

In today's industry, increasing the production capacity for an integrated paper mill is not easily accomplished. Taking into account the restrictions and regulations that exist for pulp mills, and the dollar value of additional pulping equipment, many companies are finding it advantageous to supply additional demands for pulp [to the paper mill] with recycled fibers. In addition, legislation is demanding an increase in the use of recycled fibers in paper. Therefore, the paper industry must develop methods to achieve maximum strength from an inferior furnish. Because of cost savings and fiber quality, Old Corrugated Containers [OCC] are often used as a supplement to virgin pulp. Characteristically, this furnish is predominantly softwood fibers which are longer and stronger than hardwood fibers. This feature makes OCC more appealing than other types (or grades) of recycled furnishes for addition to virgin fibers.

One drawback to using recycled OCC is that the corrugating medium found in this furnish has certain characteristics which are not desirable for addition with virgin pulp. The majority of the fibers in the medium are hardwoods which are shorter and more brittle than softwoods. Therefore, hardwoods do not provide quite as much internal strength as softwoods. In addition, the corrugating medium contains an appreciable amount of lignin which interferes with fiber bonding. Consequently, the resulting paper web's strength is decreased.

This study was undertaken to investigate the effects of pH and soaking time on the recycling of OCC and to identify the optimum repulping conditions. It is my assumption that repulping OCC under alkaline conditions will result in strength increases in the final sheet due to an increase in secondary fiber swelling and a decrease in lignin content. The increased swelling should provide increased flexibility and, in turn, a decrease in fiber cutting during processing. A decrease in the amount of refining required should be experienced due to the increased fiber swelling and flexibility. The possibility of improved cleaning efficiencies also exists due to easier fiber separation. In addition, the results will be useful in determining the need for a soaking tower when utilizing the caustic soaking technology. The benefits to industry will be a less expensive method of increasing the capacity of the pulp mill by metering in OCC *instead* of acquiring additional pulping equipment for processing virgin fibers. If results lead us to believe that repulping in an alkaline medium is optimum, an additional benefit to Kraft pulping mills is the possibility of simply adding white liquor [from the pulping process] to the OCC stock prep system in order to achieve alkalinity.

Chapter 2: Theoretical Discussion

The drying of virgin fibers causes the cell walls to collapse. This results in an increase in hydrogen bonding of the paper web. (1) The swelling ability of these fibers during recycling is reduced because the existing hydrogen bonds are not all broken. Therefore, these fibers will be less flexible and less able to form as many fiber-to-fiber hydrogen bonds when a new sheet is formed. This reduction in hydrogen bonding reduces the strength of the newly formed sheet. Other impacts that recycling has on fibers is a decrease in fiber length and an increase in total fines content. The overall reduction in fiber length often decreases the strength properties of the sheet as well. The increase in fines content does not significantly impact the strength, but does significantly impact the freeness of the stock -- which in turn impacts the drainage properties. Processes that promote fiber swelling should improve the strength potential of the pulp. The fibers should become more flexible as they swell and the development of fines during recycling should in turn be reduced due to a reduction in fiber cutting. (2) Fiber swelling is known to be improved by the use of sodium hydroxide -- therefore it could possibly enhance strength.

The effects of chemical treatments on sheet properties of recycled OCC was studied by the Institute of Paper Chemistry (3). By improving delignification, fiber swelling, and fibrillation, they had hoped to improve the bonding potential of the fibers. They tried soaking the OCC in 3% to 5% NaOH (sodium hydroxide) for 2 hours at 92 °C. Their results showed only a slight improvement regardless of the pressure applied during soaking. The OCC soaked in 5% NaOH only increased the tensile strength by 10 - 15% and did not significantly impact the burst strength.

However, they did discover an increase in freeness, burst, and tensile when the fines were removed from the stock. But, this came at a significant cost as the yield was decreased from 90% to 78%. In a more recent study (4), caustic treated pulp showed strength increases when it was subjected to a high shear field (HSF) treatment after soaking. This strength increase was attributed to the delamination of the fiber walls.

Japan uses a "tower system" in their caustic soaking technology. (5) This technology includes soaking coarsely disintegrated wastepaper in slightly alkaline warm water at high consistencies. The soaking aids in fiber swelling -- which increases the strength -- and fiber separation which allows for easier contaminant removal and a better overall recovery. It also reduces the energy required to complete the disintegration.

Under the assumption that fiber swelling and paper strength are related, several researchers have used fiber swelling as an explanation of the strength improvement. Literature states that the degree of swelling in chemical pulps is related to the number of carboxylic acid groups on the cellulose chain. (6) In addition, there is a decrease in fiber swelling, and a resulting decrease in strength, if the acid groups are partially blocked by esterification. This study also concluded that pulps, having been dried under acidic conditions, can recover their swelling properties when given an alkaline treatment after rewetting. Further studies stated that some of the acidic groups [on the cellulose chain] will dissociate in water, thereby releasing mobile counter-ions to the fiber's cell wall. (7) As the concentration of these ions increase, osmotic pressure draws additional water into the cell wall. The fibers swell as they gain water and will continue to swell until the pressure differential is balanced. Therefore, if the number of acidic groups is increased, the swelling increases as well. Conclusions were that any treatment which effects the ionization characteristics of the fibers will effect the swelling potential. Also, maximum swelling can be achieved by increasing the osmotic pressure thereby drawing more water into the fiber. This is accomplished with the ionization of the acidic groups -- which requires a higher pH.

Chapter 3: Experimental Procedures

Material

The secondary furnish used for this study was pre-consumer Old Corrugated Containers. This furnish [DKL clippings] was supplied by Green Bay Packaging. This furnish was chosen over a post-consumer source of OCC due to its cleanliness. The post-consumer source contained printing inks, glues, waxes, and tapings. An inability to properly remove all of these contaminants [for testing purposes] resulted in the elimination of this source from the project. Specific chemicals for pH adjustment were the following: 2% Sodium Hydroxide (NaOH) to achieve alkalinity; 2% Sulfuric Acid (H_2SO_4) to achieve acidity. Common tap water was used for all trials except where noted.

Equipment

Western Michigan University's laboratory *Morden Slushmaker* was used to repulp the secondary furnish. The laboratory *Valley Beater* was used when refining was needed. The handsheets were formed with the laboratory's *Noble and Wood Handsheet Mold* while recycling the white water. This handsheet mold makes uniform handsheets that are 12 x 12 inches. Fiber Length Classification was determined by WMU's *Kajaani FS-100* fiber analyzer. Lastly, the apparatus used to obtain the water retention value [WRV] of the pulp was a common laboratory centrifuge with a modified cup. The cup was equipped with a removable screen inside so that the water could be filtered from the stock sample. The design can be found in **Appendix III**.

Procedures

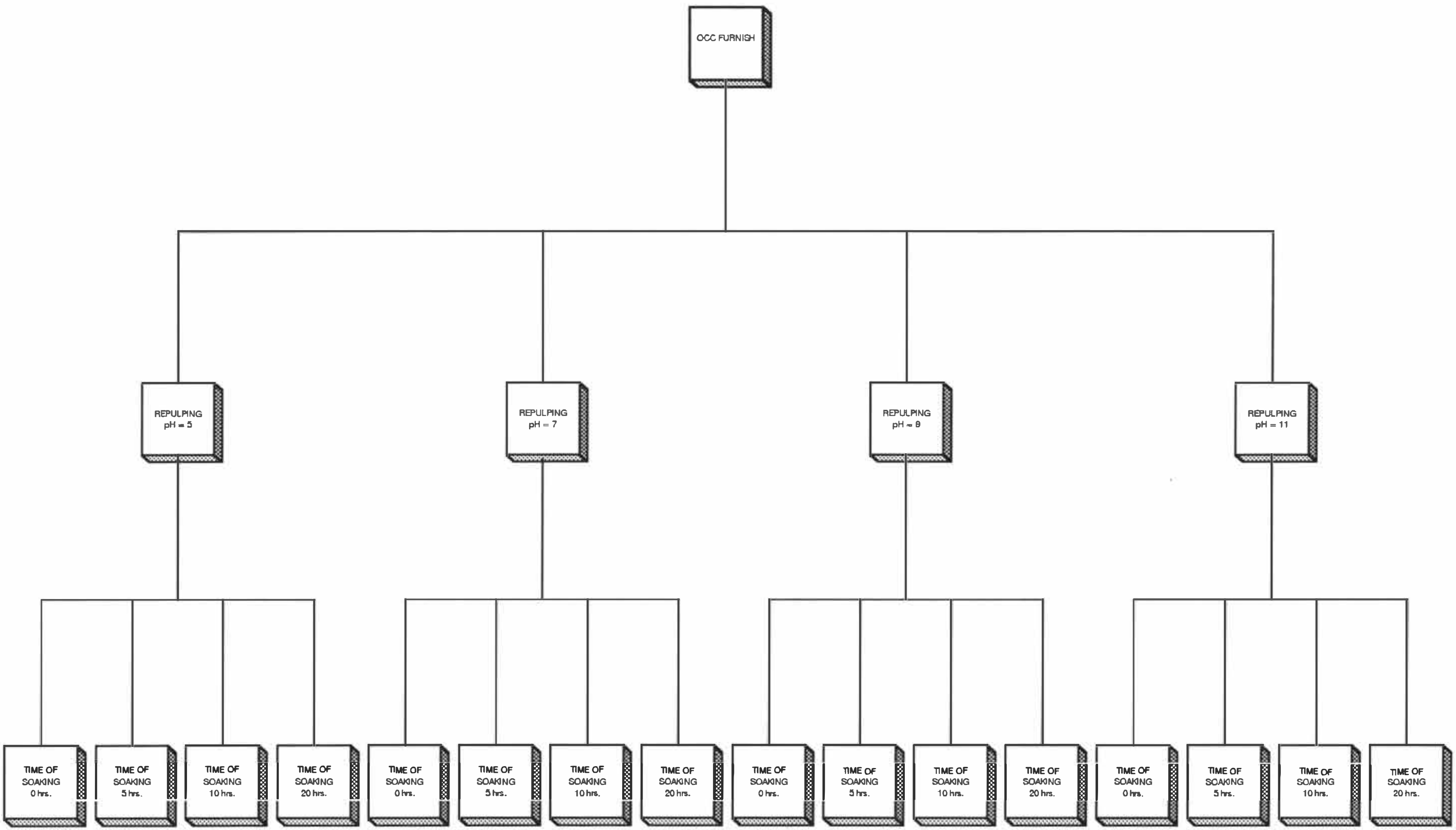
The OCC furnish was repulped in the Morden Slushmaker at 6% consistency and 65 °C for 30 minutes. Four pounds of O.D. furnish was the batch size for each pH level. Repulping occurred at four pH levels. These levels were 5, 7, 9, and 11. These levels were obtained through the addition of the above mentioned chemicals. For each pH level, the repulped stock was stored for varying lengths of time while maintaining the desired pH and temperature. Soaking times for each pH level were 0, 5, 10, and 20 hours. This resulted in 16 one-pound batches of pulp for analysis. All soaking was conducted at atmospheric pressure.

After soaking, each batch of stock was tested to determine its freeness (CSF), water retention value, and fiber length classification. Beater curves were also performed in order to determine the effects of the variables on the beating of the pulp. These curves were only performed on the samples for the 0 and 20 hour soaking times. All beater runs were adjusted to a pH of 8 immediately prior to beating. ***TAPPI*** standards were followed during all testing (where applicable).

Lastly, 2.54g handsheets [12.6 lb/MSF] were prepared in the laboratory 8 x 8 inch Noble and Wood Handsheet Mold. All handsheets were formed at a pH of 5 while recycling the white water. The handsheets were then conditioned and their physical properties tested by various ***TAPPI*** standard test methods. Test results were analyzed statistically in order to investigate the effects of pH and soaking time on the recycling of OCC and to identify the optimum repulping conditions with respect to pH and soaking time. [Please see next page for design model].

PROCEDURE ORGANIZATIONAL CHART

Effects of pH and Soaking Time on the Repulping of OCC



Testing will occur on each pulp sample of specific pH and soak time

Chapter 4: Discussion of Results

Appendix One contains the test results in spreadsheet form. Table One provides the results for the pulp samples. Table Two provides the handsheet test results. Appendix Two contains all graphs and charts used for graphical analysis of the test results. Graphs 1-17 are line graphs providing two-dimensional analyses of the results. Charts 1-6 are three-dimensional contour plots providing the optimum repulping conditions [with respect to pH and soaking time] for strength improvement in OCC.

Freeness and Fiber Classification

As seen in Graph 1, increasing the pH during repulping resulted in a corresponding decrease in the freeness of the stock after repulping. Significant decreases in the freeness were noted as the pH was raised from 5 to 7 and then to 9. The freeness after repulping at a pH of 11 was not significantly different from the freeness at pH 9. This decrease in freeness may have occurred due to an increase in the fines content of the pulp resulting from fiber cutting during repulping. Another explanation is that there was an increase in fiber swelling thereby increasing the "water holding" ability of the pulp. Increasing the soaking time after repulping did not significantly affect the freeness of the pulps.

Graphs 2-4 show the effects of pH and soaking on fiber length. The average fiber lengths for the batches repulped at pH's of 5, 7, and 9 were not significantly different. However, the pH of 11 had a significantly lower average fiber length. [See Graph 2] At this time, the reason for the decrease in average fiber length for pH 11 in not known. Again, the increase in soaking time did not seem to significantly affect the results. The results for the % fines content and % long fiber portion of the pulps support the average fiber length data. There were no significant differences for the pH's of 5, 7, and 9. The soaking times showed no significant differences either. But, the batch at pH 11 had a significantly greater percentage of fines and lower percentage of long fiber. Therefore, the fibers repulped at pH 11 experienced a reduction in fiber length. This would explain the lower freeness values obtained from this batch, but does not explain why the freeness decreased for every increase in pH.

Water Retention Value and Apparent Density

Water Retention Value [WRV] is used as an indication of fiber swelling. This test measures the amount of water (by weight) contained in a pulp sample after centrifuging for 30 minutes at a force of 900g. The test result is calculated in grams of water per gram of fiber. An increase in the WRV indicates an increase in fiber swelling.

Graph 5 shows the WRV results of the pulp. Immediately after repulping [soaking time = 0], there was no significant difference in WRV for the 4 pH levels. As the soaking time was increased, the WRV showed significant differences. The WRV for the batches at pH 9 and 11 both increased as soaking time increased. This indicates that the fibers swelled and gained water as they soaked. The neutral batch [pH = 7] did not change with soak time. The batch at pH 5 showed the opposite: as the soaking time increased, the WRV decreased meaning that the fibers actually constricted or became smaller across their diameter resulting in less water being held internally by each fiber. These results are a better explanation for the freeness trends. As the WRV and swelling increased, the freeness of the pulps decreased. These results indicate that repulping under alkaline conditions should increase the fibers flexibility and possibly the resulting strength of the handsheets.

Graph 6 shows the effects of pH and soaking on the apparent density of the handsheets. As suggested earlier, we see an increase in the density of the handsheets as the pH during repulping was increased. The pH 5 sheets were the least dense. The sheets for the batches at 7 and 9 were higher, but not significantly different from each other. Lastly, the pH 11 pulp produced a significantly denser sheet. However, a portion of this increase can be attributed to the shorter fiber lengths and increased fines content of this batch.

To summarize, as WRV and swelling increased, the density of the handsheets increased as well. The fibers appear to be more flexible and more able to conform to the new sheet. This will provide for a greater contact area between the fibers in the sheet resulting in stronger bonds and a possible increase in the number of bonds in the sheet. Therefore, as pH is increased we should see an increase in the strength of the handsheets -- except possibly for the pH 11 handsheets due to their lower fiber lengths.

Strength Properties of Handsheets

Graphs 7 - 11 show the strength results for the handsheets. As pH was increased from 5 to 7 and then to 9, we see significant increases in all the strength properties tested. These tests being:

Tensile Index, TEA Index, % Stretch, Burst Index, and Zero-Span Tensile Strength. The results for the batch at pH 11 fell near the pH 7 or 9, depending on the test. As mentioned earlier, the pH 11 sheets were not as strong due to the shorter fiber content. The zero-span results [graph 11] for the pH 11 sheets were the same as pH 9 indicating that the internal strength of the fibers were similar, but the shorter average length resulted in a slightly weaker sheet.

The soaking time also affected the strength results. As the soaking time was increased to 5 hours, an increase in the strength of the handsheets was obtained. The results after 10 hours of soaking were not significantly different from those after 5 hours of soaking. Lastly, after 20 hours of soaking, the strength results decreased back to -- or below -- that of the 0 hour batch. The effects of soaking time were more evident in the alkaline batches. One notable exception is that the soaking time did not significantly affect the Burst Index of the handsheets. Increases in the Burst Index of the handsheets is attributed ***only*** to the increase in pH during repulping.

Beating of the Fibers

Graphs 12 and 13 show the effects of pH and soaking time on the beating of the pulp batches. As seen in these two graphs, the freeness decreased with increased beating -- as expected -- but repulping at different pH's did not affect the way the pulp "handled" the beating. These two graphs are almost identical indicating that the soaking time also had no effect on the beating of the fibers with respect to freeness. Graph 14 indicates that neither the pH nor the soaking time affected the % fines of the pulp during beating. The % fines did not change significantly during beating indicating that there were no fines being formed during beating -- regardless of the pH during repulping. The % fines for the batch at pH 11 is significantly higher than the rest, but this was seen in Graph 3 and is not due to the beating of the pulp. Similar results were obtained for the % long fiber and average fiber length.

Chapter 5: Conclusions and Recommendations

Increasing the pH during repulping increases the secondary fiber swelling. This increased swelling increases the flexibility of the fibers so that they are more conformable to the newly formed sheet. A more dense sheet is formed as a result of the cell walls being more collapsible. This provides a greater bonding area between fibers and also the possibility of increased bonding. As the number of bonds, and the strength of each bond, both increase; so does the resulting strength of the sheet.

Charts 1 through 6 in Appendix Two are three-dimensional plots of the strength results showing the optimum repulping conditions. The two x-axes are pH and soaking time, the y-axis is the strength property. Tensile Index, TEA Index, and % Stretch all have maximum results at a pH of 9 and 5 to 10 hours soaking time. At these conditions, the increases over the neutral batch [pH = 7] were 11%, 19%, and 5%, respectively. The increases were even greater over the results for the pH 5 batch.

Burst Index, as previously mentioned, was not affected by the soaking time. The maximum seen in Chart 5 was also at a pH of 9. This was a 10% increase over the neutral batch. The Zero-Span Tensile Strength plot [Chart 6] showed an absolute maximum between the pH's of 9 and 11 and slightly over 10 hours of soaking. However, the optimum conditions have been determined to be at a pH of 9 and 5 to 10 hours soaking. At these conditions, the Zero-Span maximum had a 3% increase over the neutral batch.

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APPENDICES

APPENDIX ONE

TABLE ONE: PULP PROPERTIES

Property	Units																
pH	pH	5	5	5	5	7	7	7	7	9	9	9	9	11	11	11	11
Soak Time	hours	0	5	10	20	0	5	10	20	0	5	10	20	0	5	10	20
Freeness:																	
0 min	ml C.S.F.	559	574	575	571	548	538	552	566	517	529	530	532	513	527	524	525
15 min	ml C.S.F.	406			438	428			385	417			424	408			422
30 min	ml C.S.F.	377			367	378			360	358			373	355			352
45 min	ml C.S.F.	284			302	294			297	291			332	267			306
WRV	g water/g fiber	2.27	2.23	2.18	2.10	2.26	2.25	2.26	2.27	2.25	2.32	2.33	2.32	2.30	2.39	2.42	2.44
Fiber Classification																	
Average Fiber Length:																	
0 min	mm	1.63	1.71	1.70	1.62	1.70	1.74	1.73	1.71	1.71	1.78	1.81	1.76	1.44	1.48	1.54	1.51
15 min	mm	1.71			1.83	1.84			1.84	1.74			1.82	1.52			1.54
30 min	mm	1.69			1.79	1.74			1.93	1.88			1.75	1.40			1.53
45 min	mm	1.85			1.83	1.79			1.85	1.83			1.75	1.45			1.57
% Fines: [< 0.2mm]																	
0 min	%	25.59	25.10	24.98	25.48	24.67	23.67	23.79	24.12	27.28	24.28	23.05	24.66	29.12	28.64	29.33	30.76
15 min	%	25.10			21.53	19.93			21.45	34.88			22.51	30.92			30.93
30 min	%	22.57			22.96	24.92			22.23	21.96			22.24	28.59			31.02
45 min	%	23.62			20.42	*****			22.95	19.19			23.21	29.57			31.51
% Long Fiber: [> 0.45 mm]																	
0 min	%	64.17	66.04	66.88	65.31	66.21	67.35	67.18	67.49	64.55	67.10	68.76	66.66	58.05	60.79	59.82	60.24
15 min	%	66.31			70.10	72.12			69.81	56.09			69.06	59.57			60.93
30 min	%	68.82			69.65	66.23			70.50	70.50			69.20	58.44			59.87
45 min	%	68.61			71.68	*****			69.95	71.76			67.73	59.71			61.07

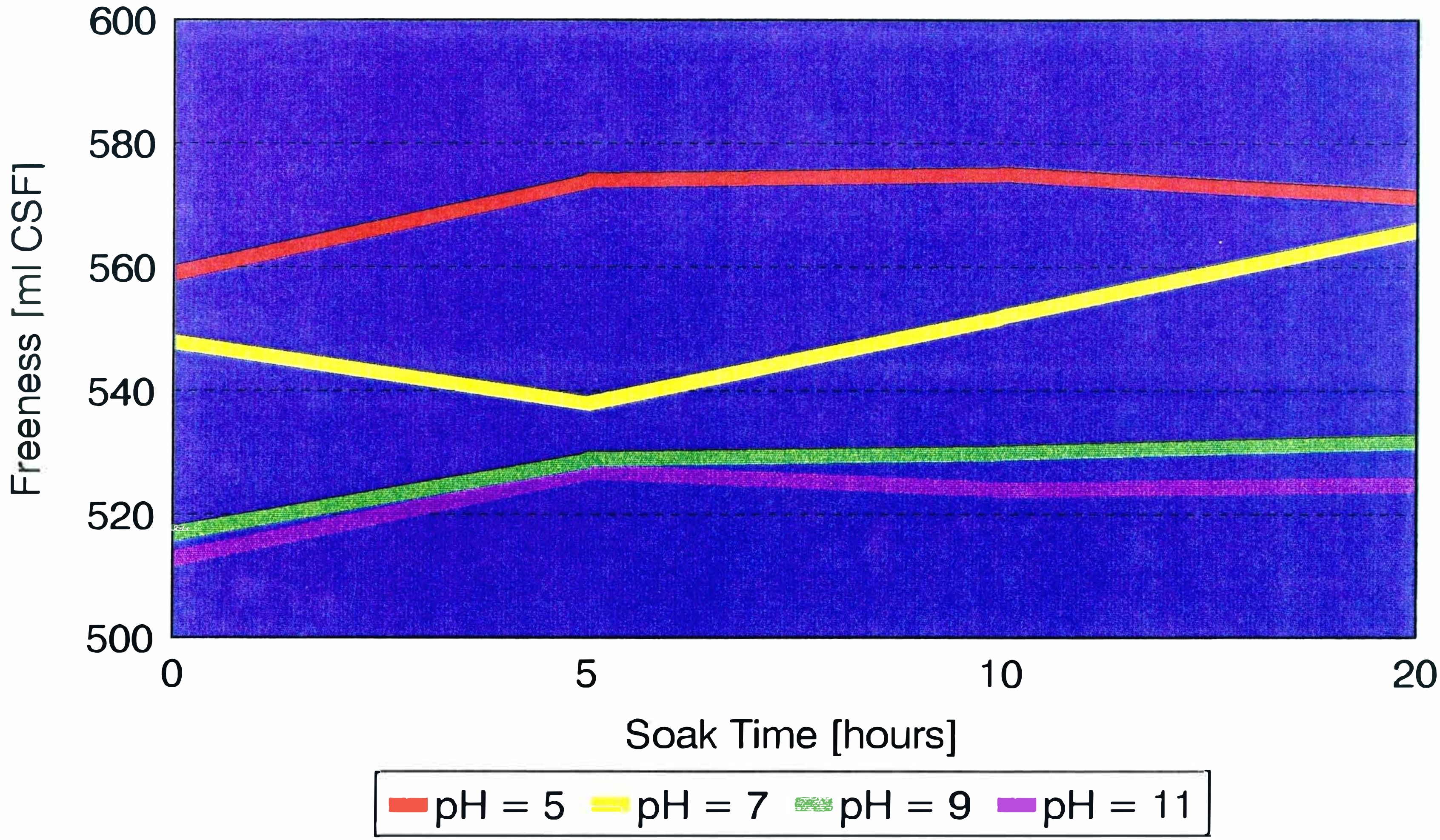
TABLE TWO: HANDSHEET PROPERTIES

Property	Units																
pH	pH	5	5	5	5	7	7	7	7	9	9	9	9	11	11	11	11
Soak Time	hours	0	5	10	20	0	5	10	20	0	5	10	20	0	5	10	20
Grammage	g/sq meter	63.65	59.60	63.40	63.04	64.11	63.36	63.36	63.36	61.25	64.20	65.19	65.03	63.40	64.11	64.73	64.96
Caliper	points	6.44	6.37	6.70	6.68	6.42	6.45	6.46	6.47	6.32	6.46	6.54	6.56	6.24	6.12	6.21	6.24
Density	g/cc	0.383	0.368	0.372	0.372	0.393	0.387	0.386	0.386	0.389	0.391	0.392	0.390	0.400	0.412	0.411	0.410
Tensile Index	Nm/g	31.19	30.87	31.43	30.61	32.25	33.88	33.12	32.18	34.40	36.49	36.40	34.32	35.05	35.32	34.76	33.94
TEA Index	mJ/g	710	685	687	676	755	793	812	665	926	947	962	897	910	903	885	768
Stretch	%	2.18	2.15	2.18	2.16	2.16	2.31	2.29	2.23	2.27	2.37	2.44	2.32	2.22	2.25	2.23	2.18
Burst Index	kPam2/g	1.54	1.38	1.48	1.44	1.92	1.84	1.81	1.79	2.04	2.04	2.02	2.03	1.98	1.83	1.76	1.80
Zero-Span Tensile	kg/15mm	10.99	10.48	10.72	10.84	11.11	11.38	11.46	11.44	11.34	11.65	11.71	11.63	11.51	11.58	11.84	11.65

APPENDIX TWO

Graph 1

Soak Time vs. Freeness



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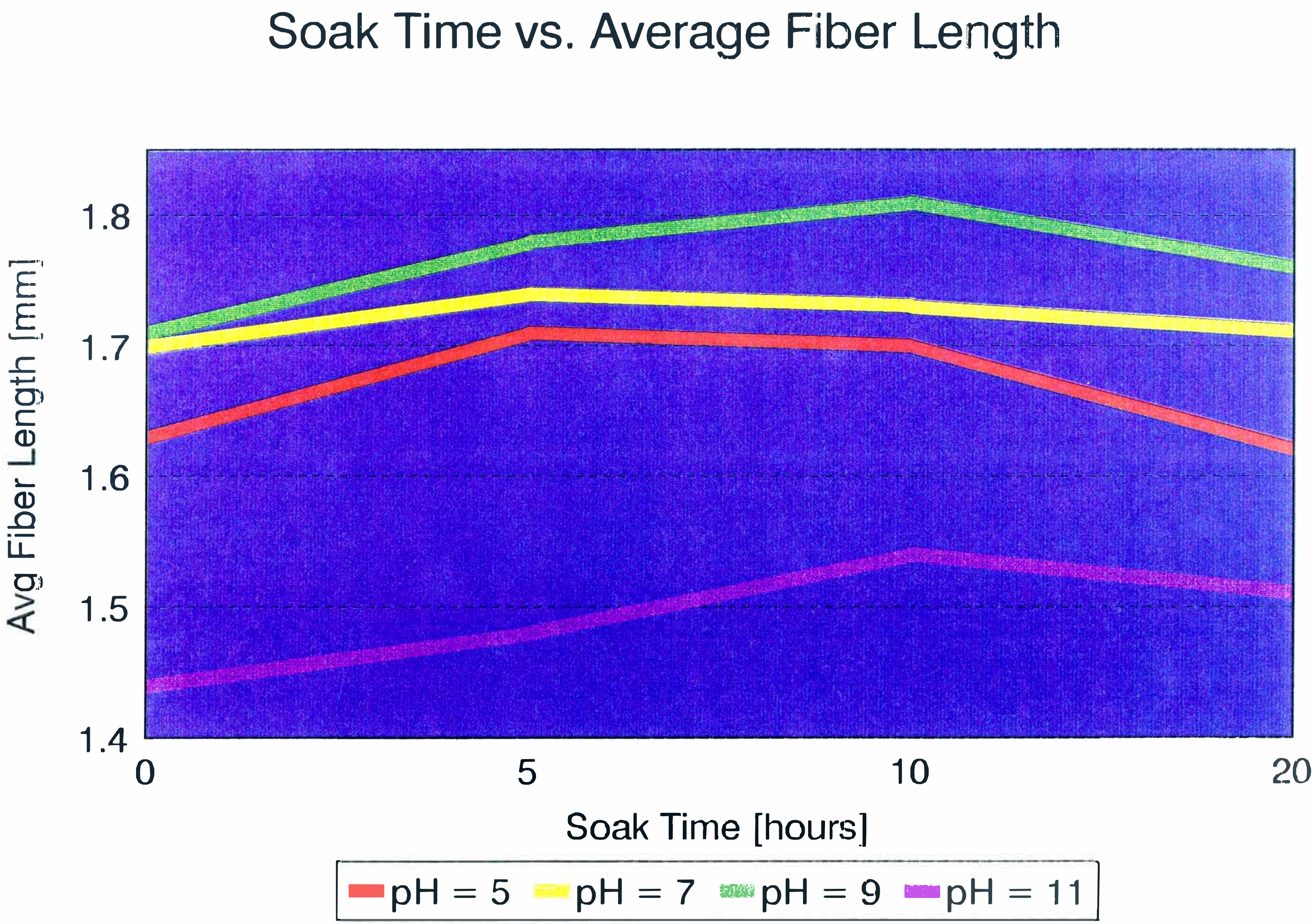
Graph 2



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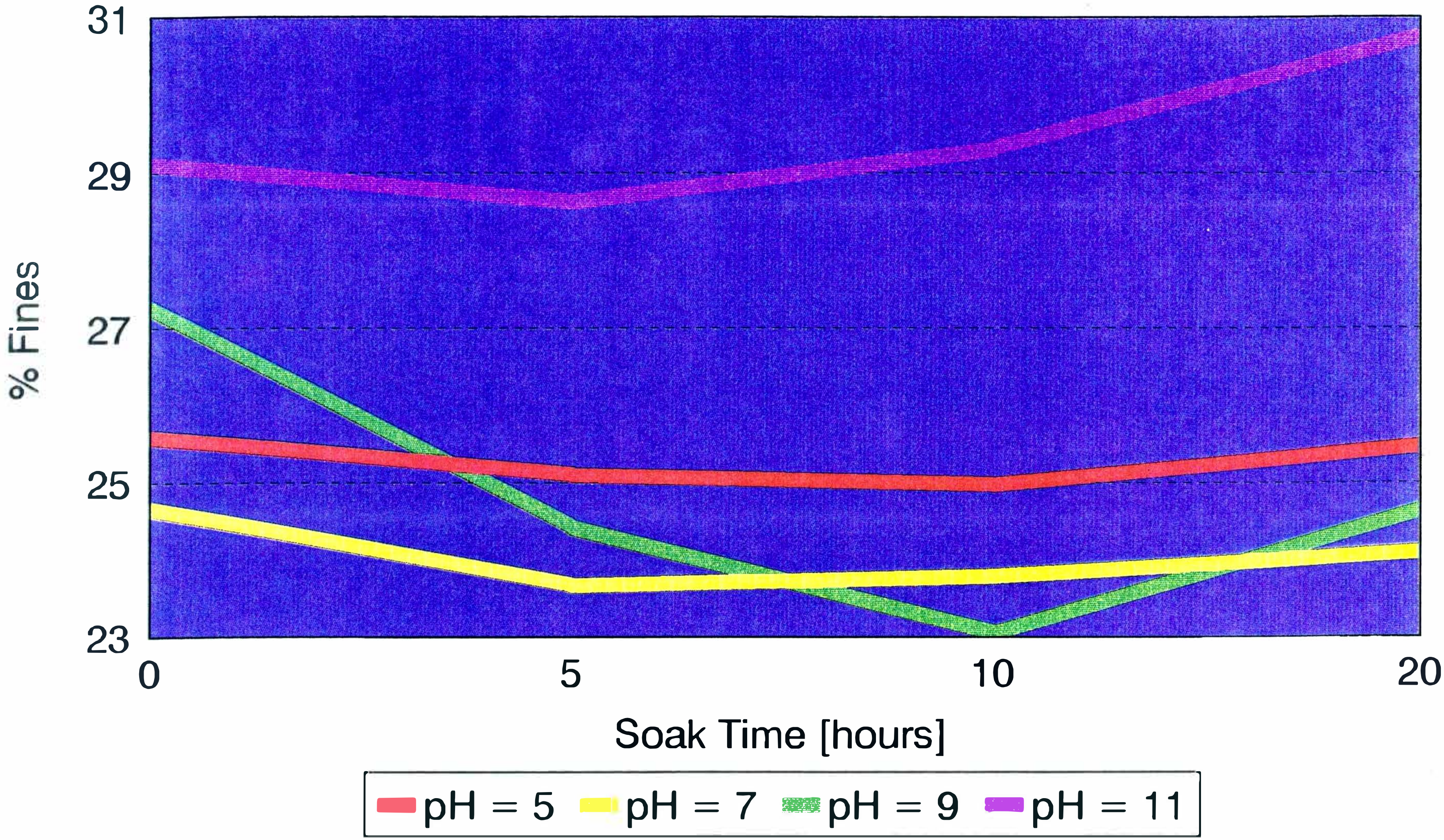
Graph 3

Soak Time vs. % Fines



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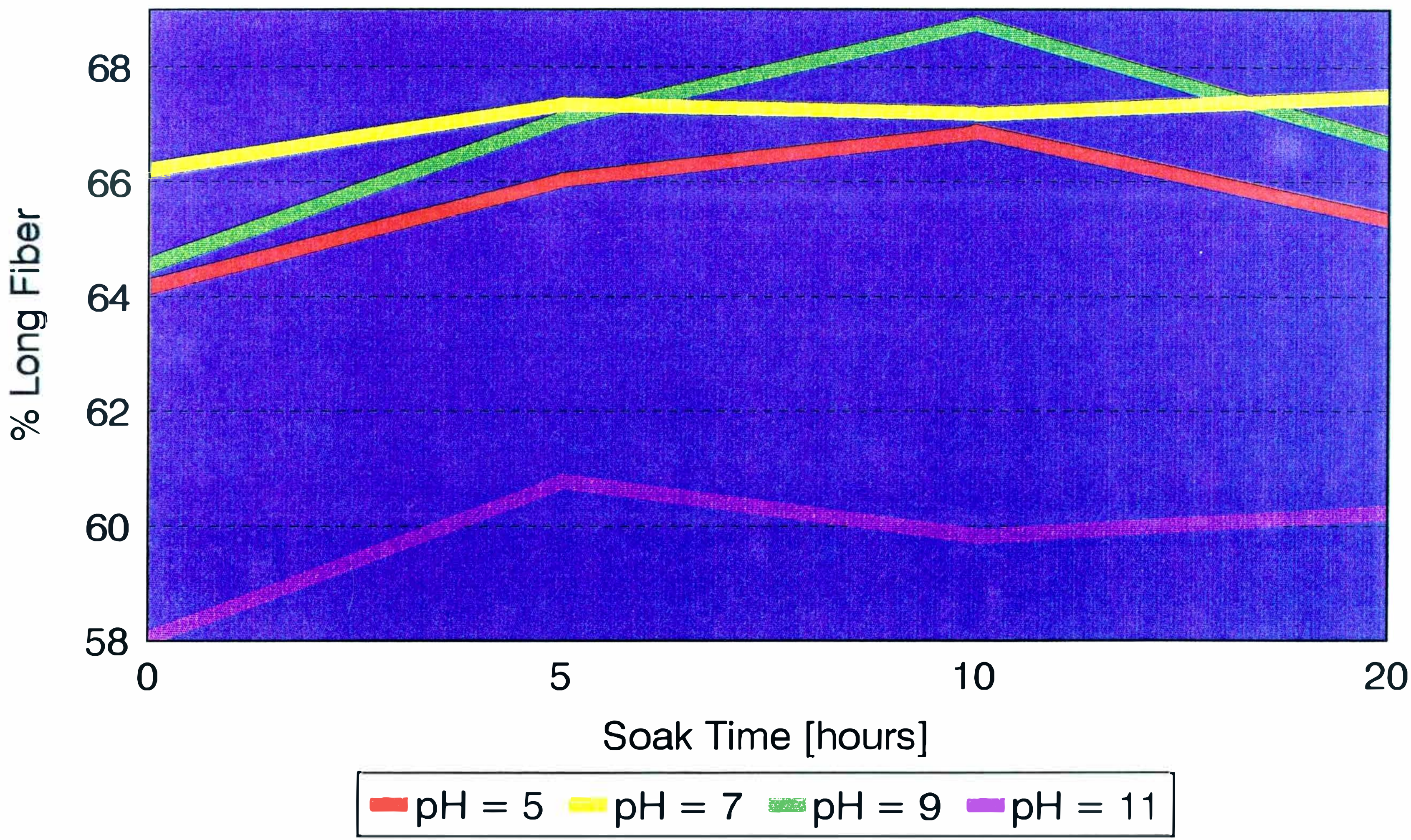
Graph 4

Soak Time vs. % Long Fiber



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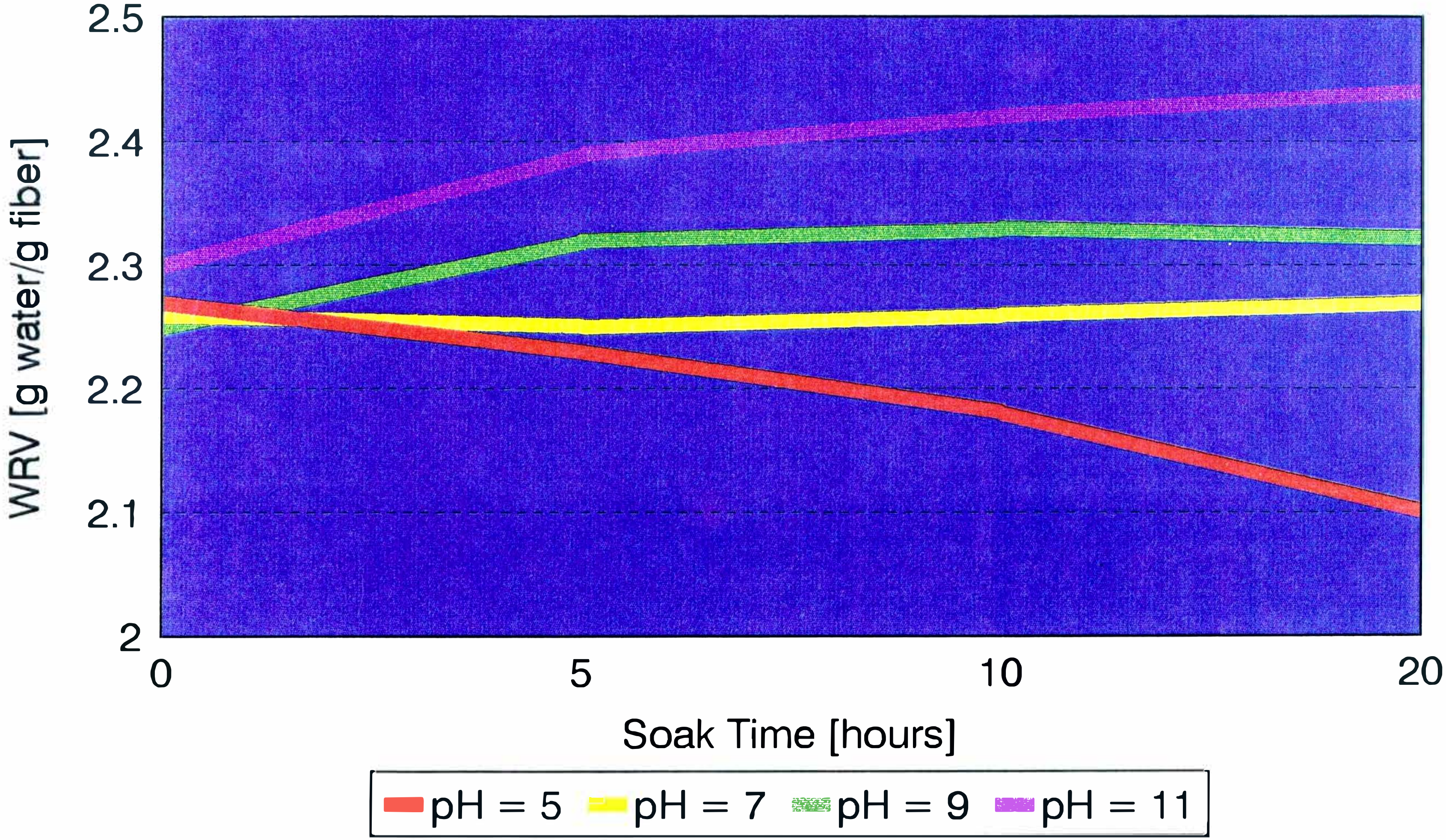
Graph 5

Soak Time vs. Water Retention Value



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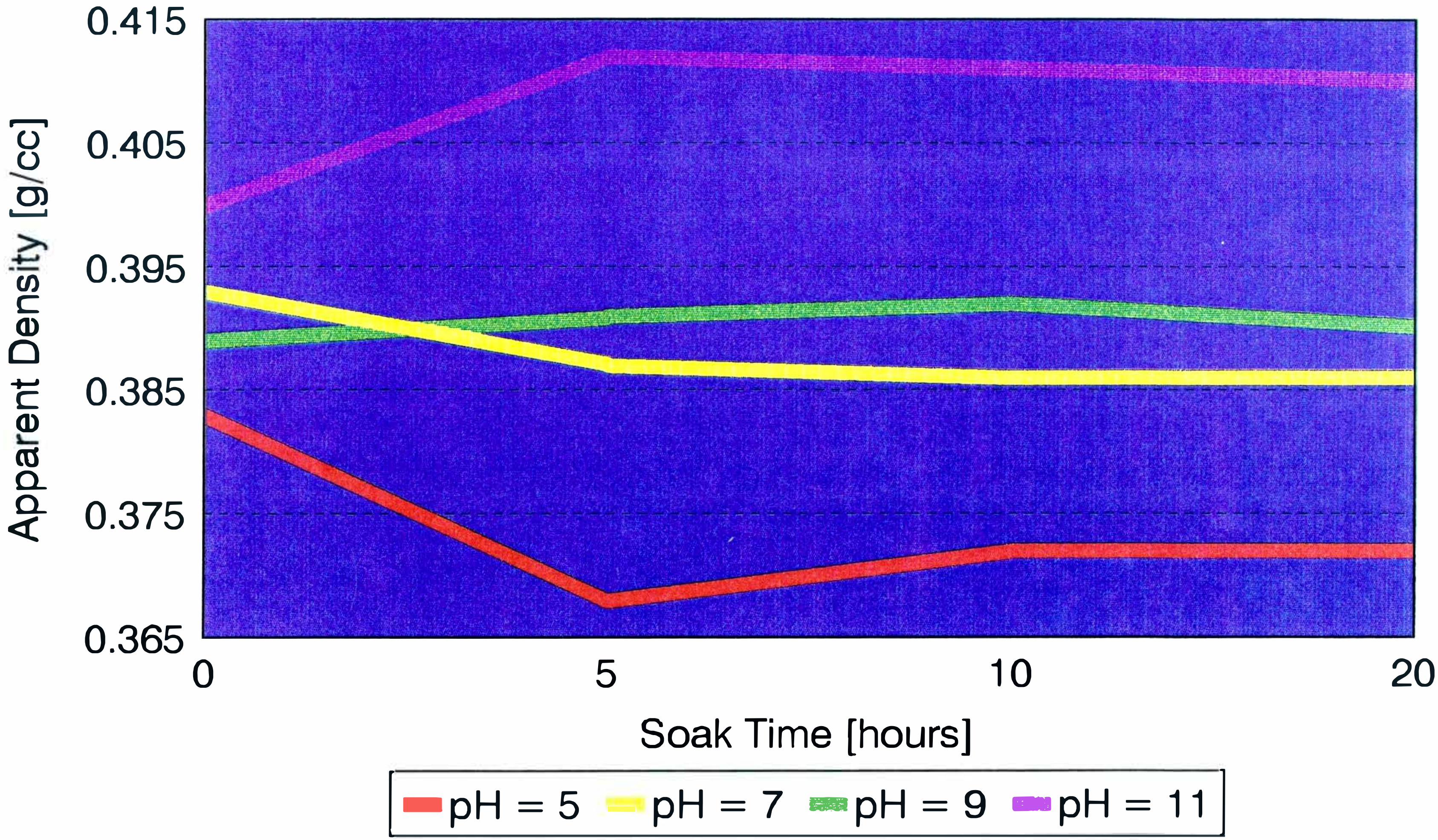
Graph 6

Soak Time vs. Apparent Density



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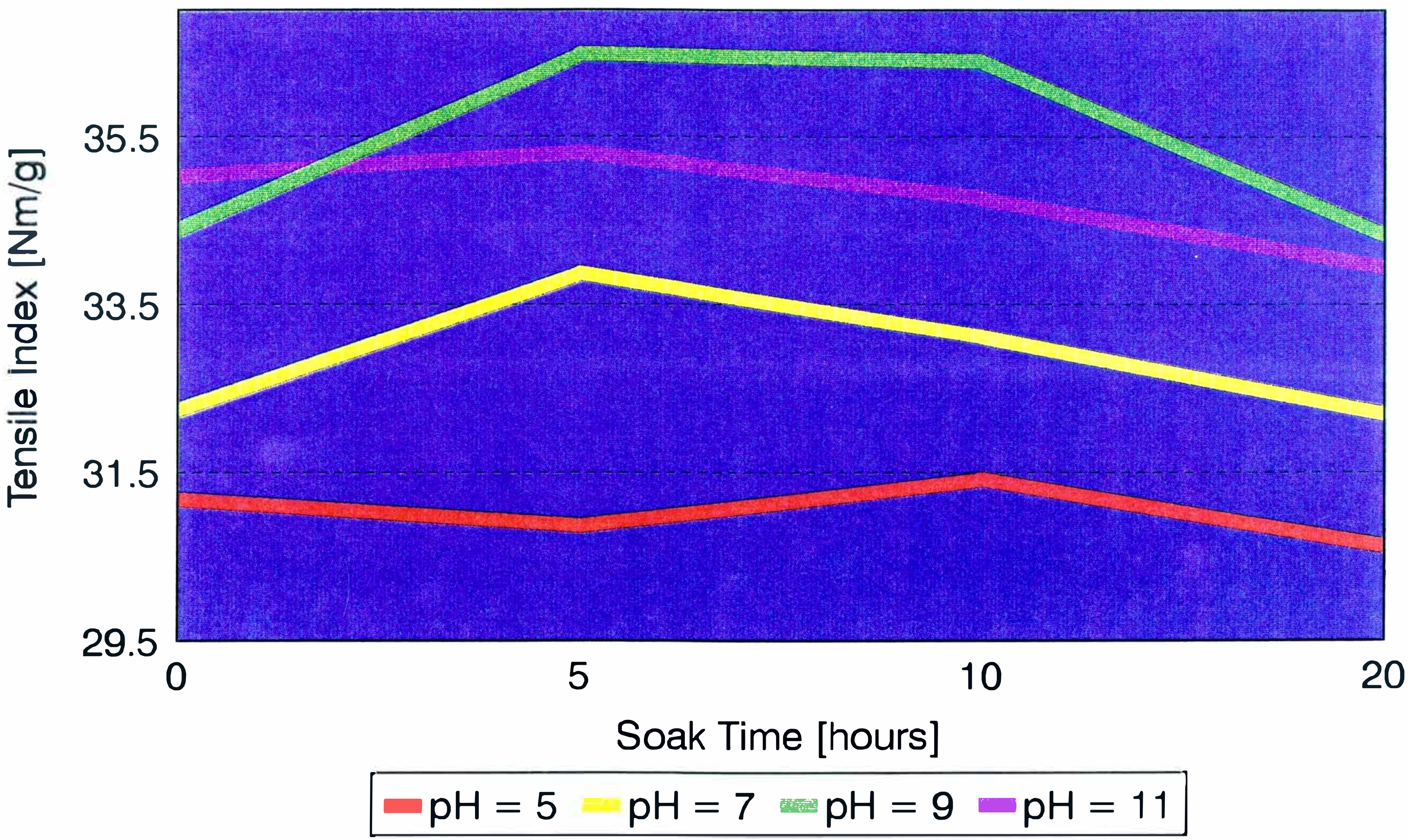
Graph 7

Soak Time vs. Tensile Index



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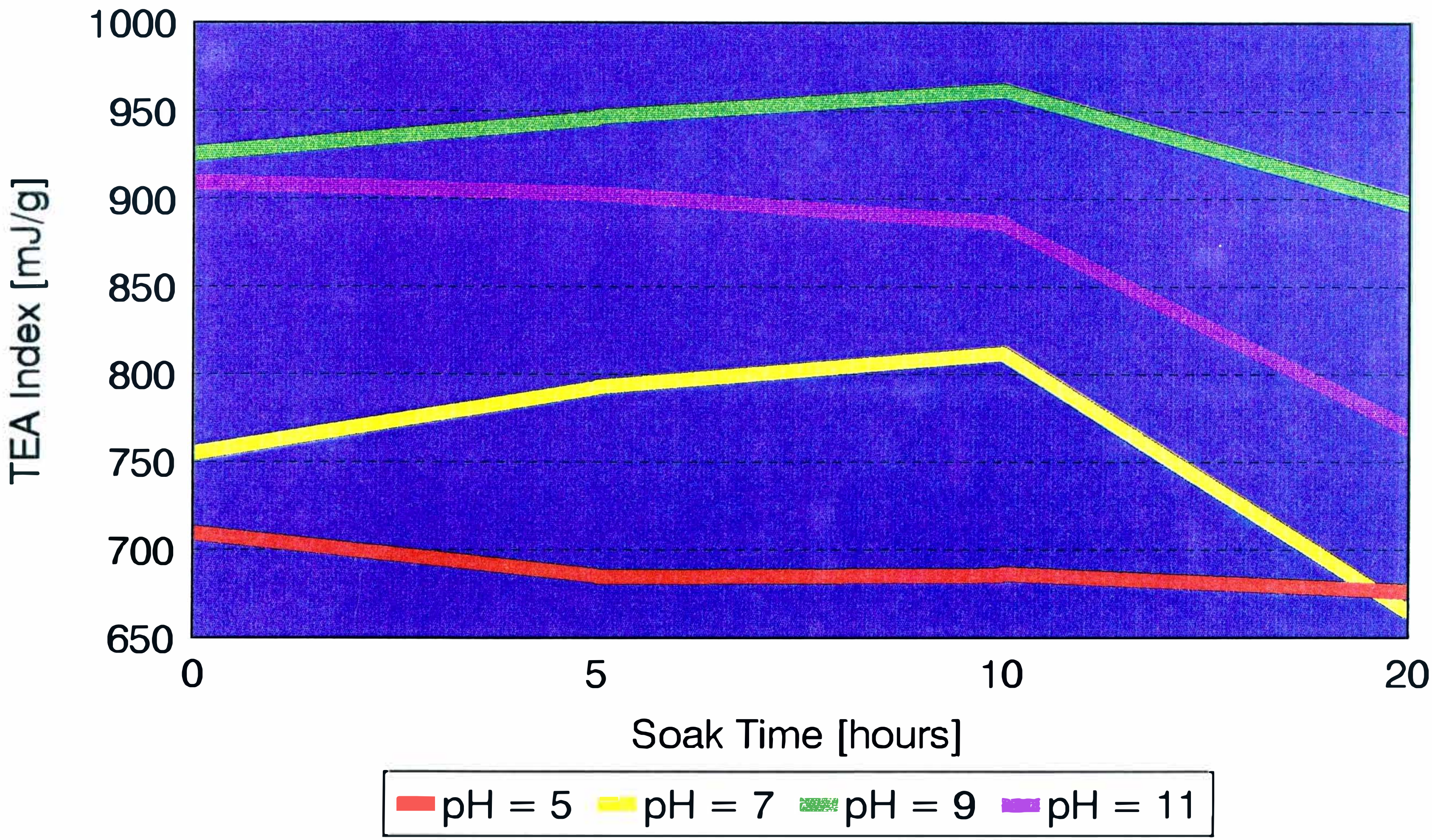
Graph 8

Soak Time vs. TEA Index



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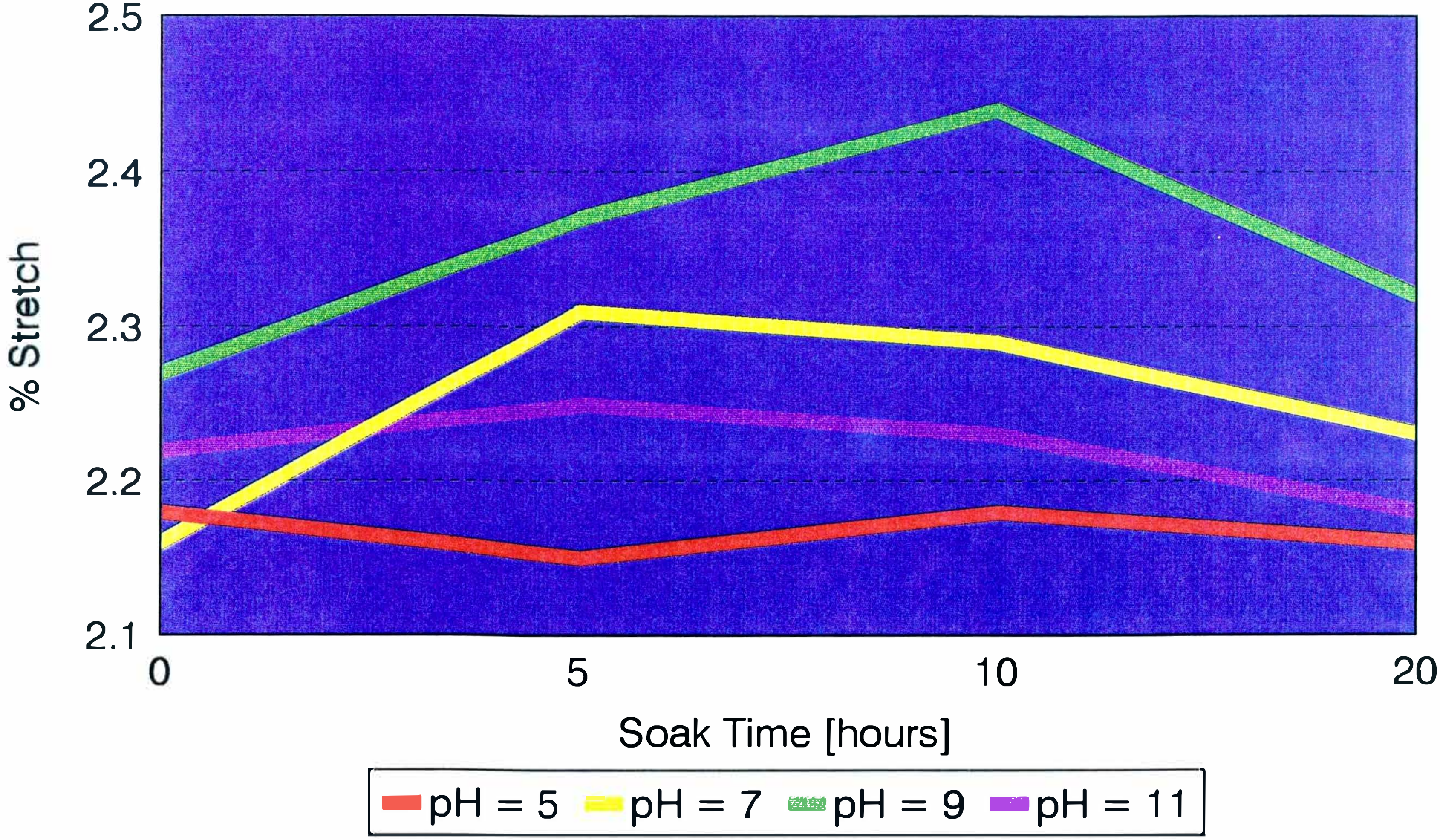
Graph 9

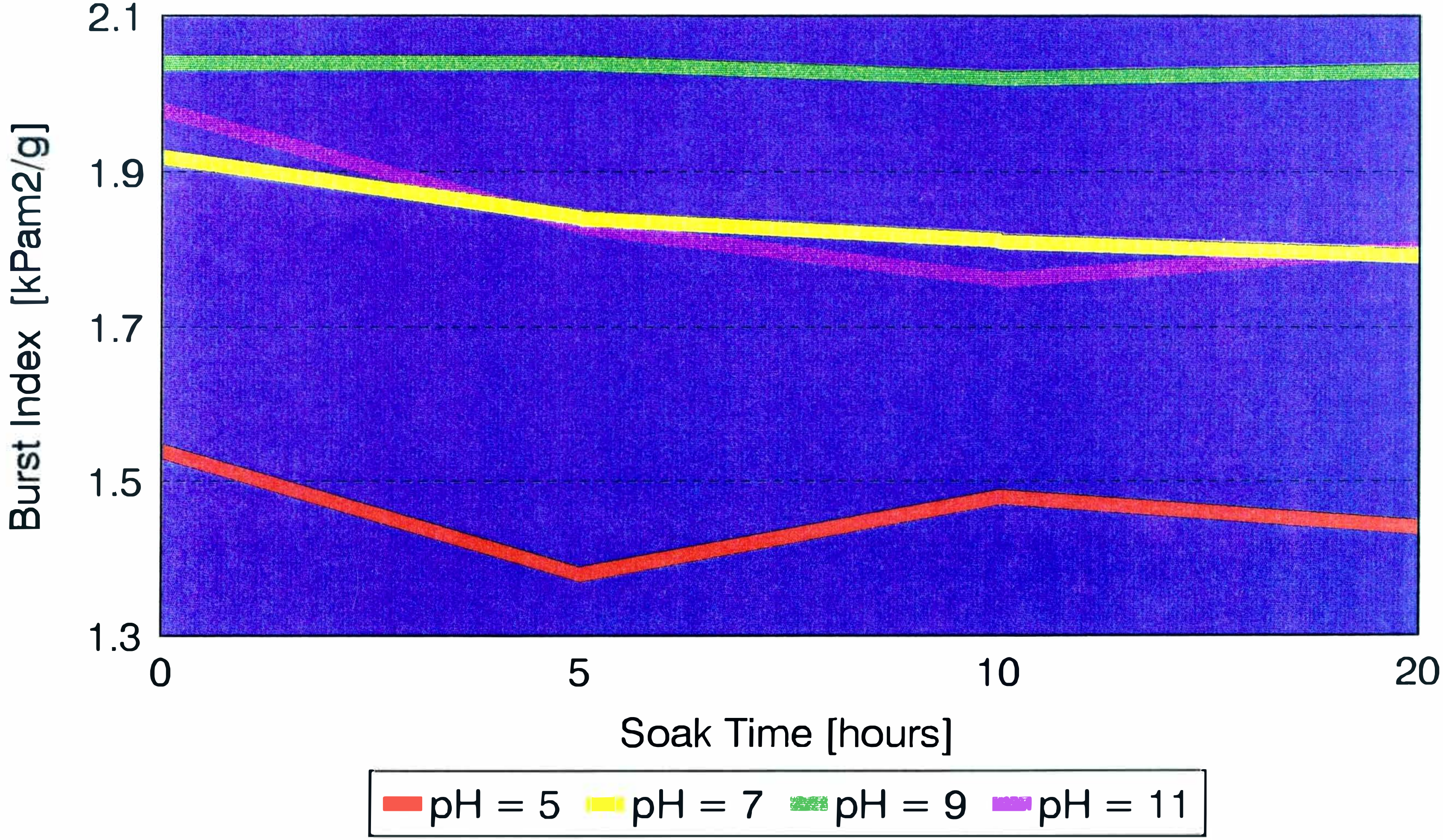
Soak Time vs. % Stretch



JSE 4-6-94

Graph 10

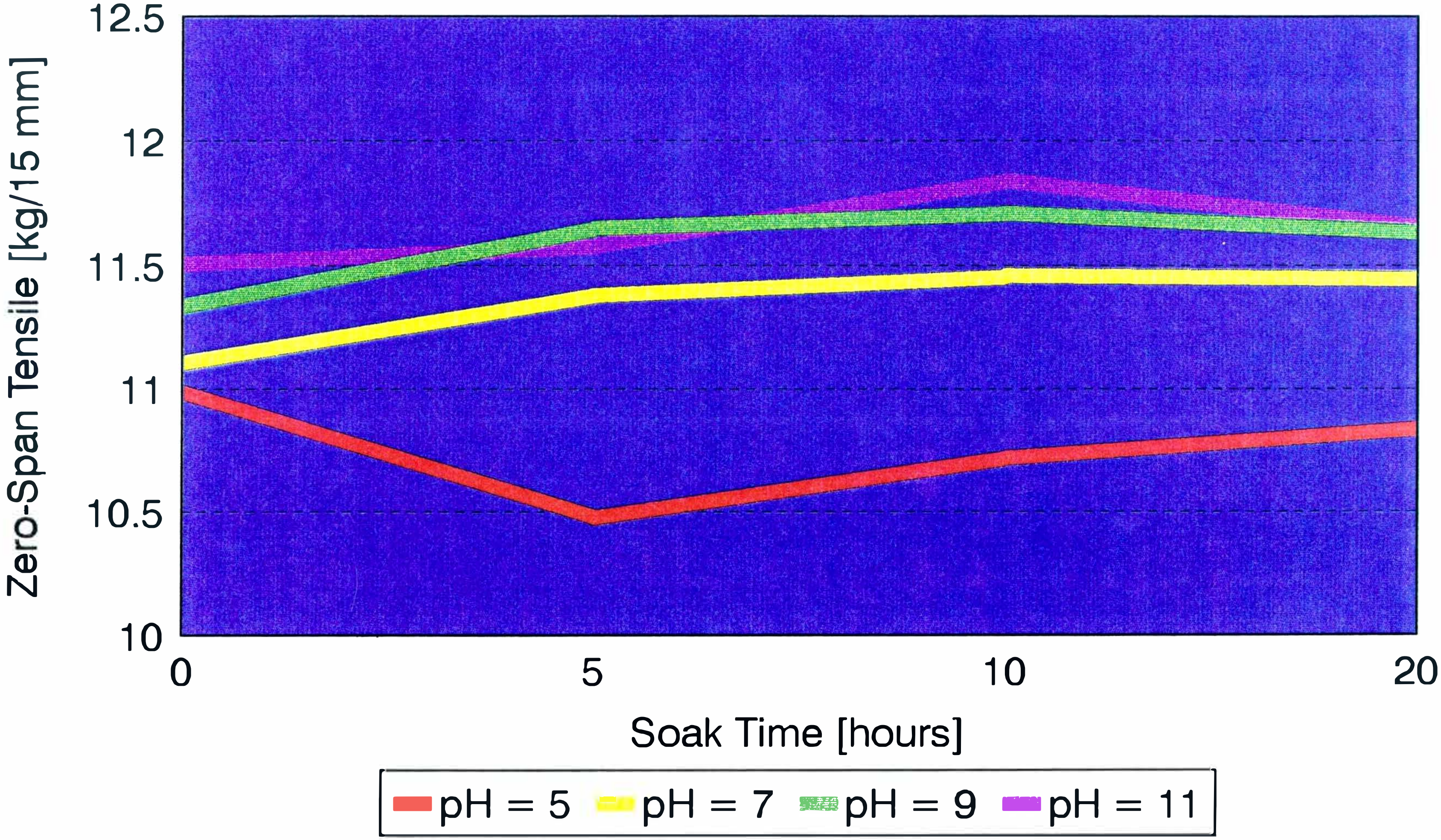
Soak Time vs. Burst Index



JSE 4-6-94

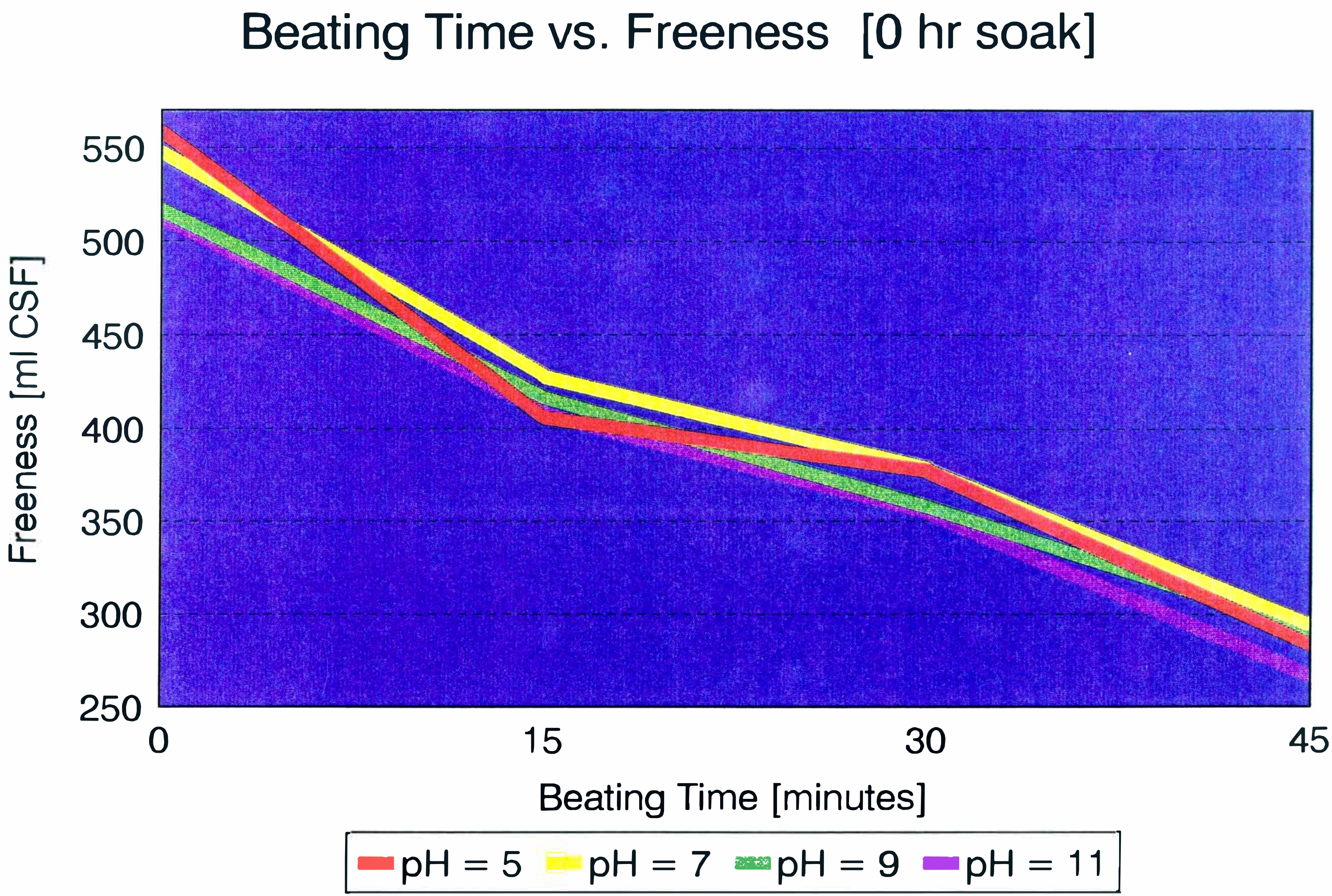
Graph 11

Soak Time vs. Zero-Span Tensile Strength



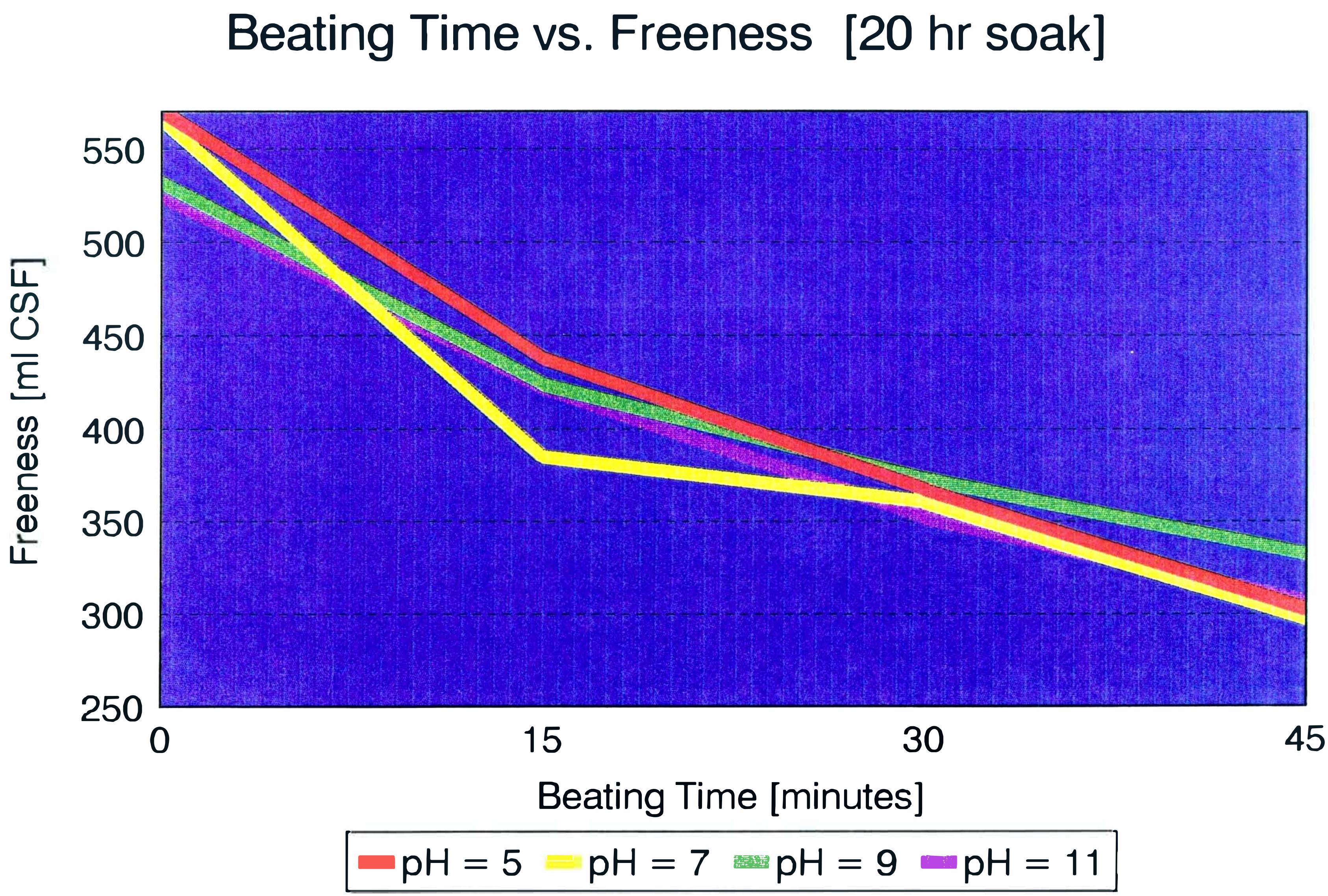
JSE 4-6-94

Graph 12



JSE 4-7-94

Graph 13



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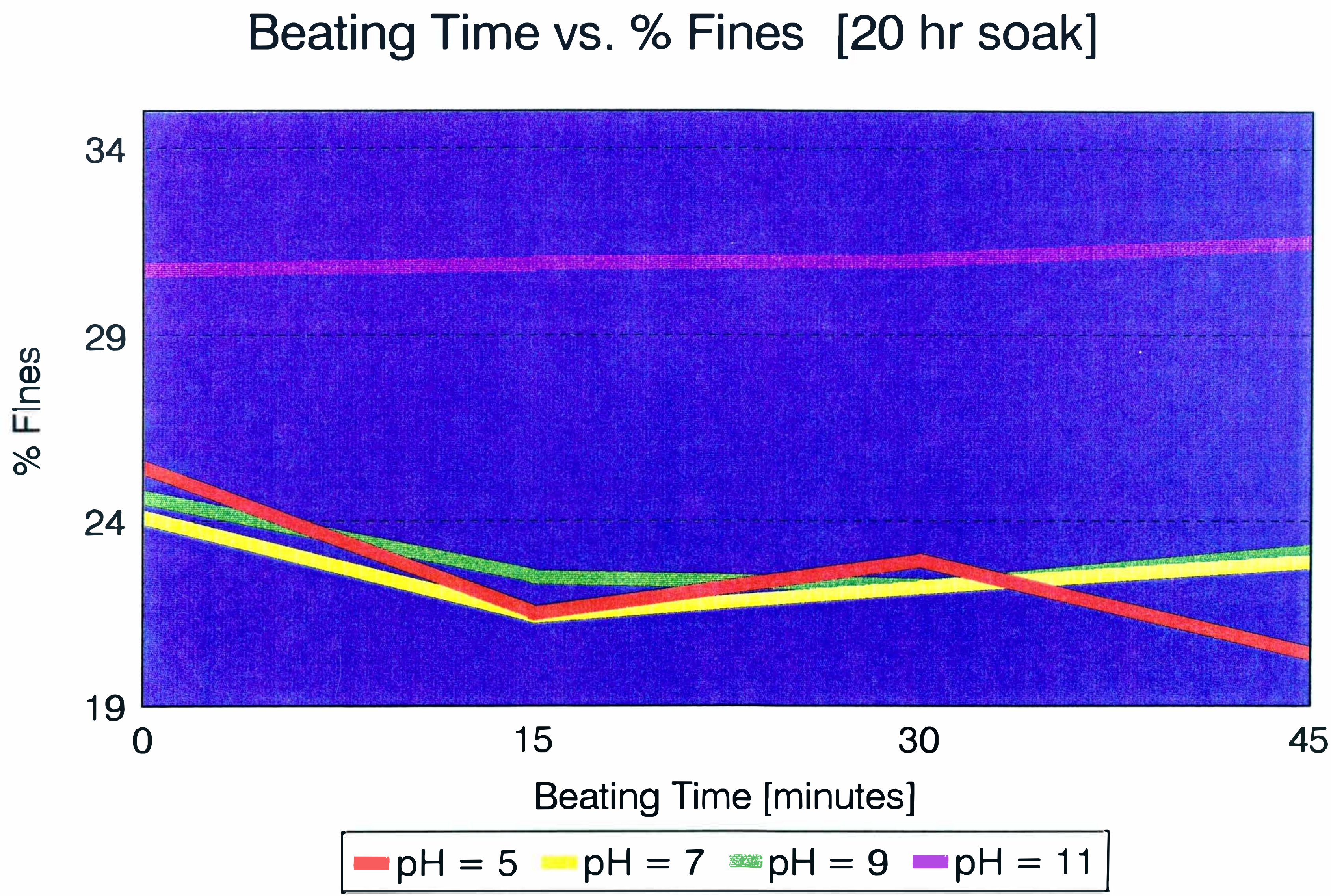
Graph 14



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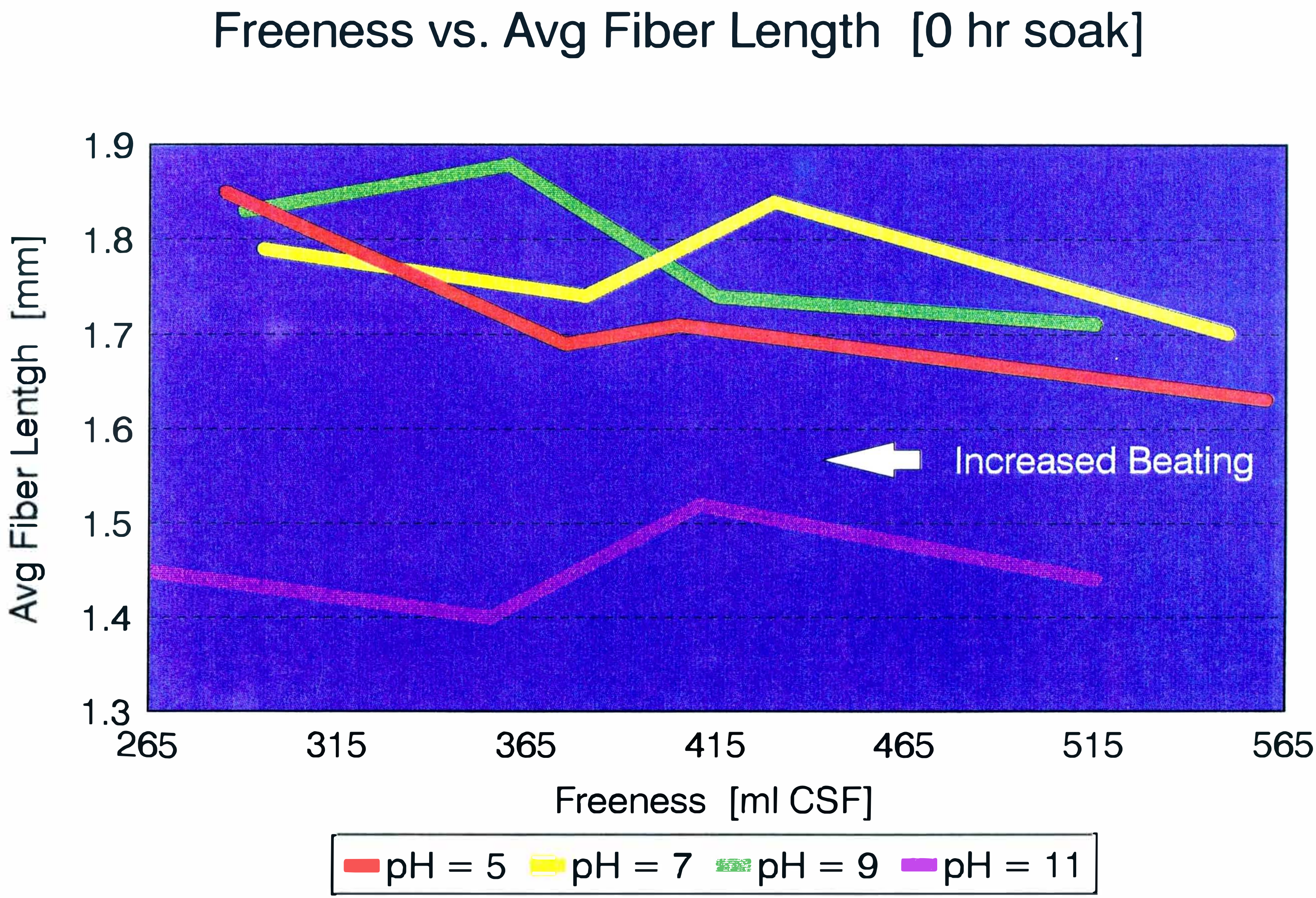
Graph 15



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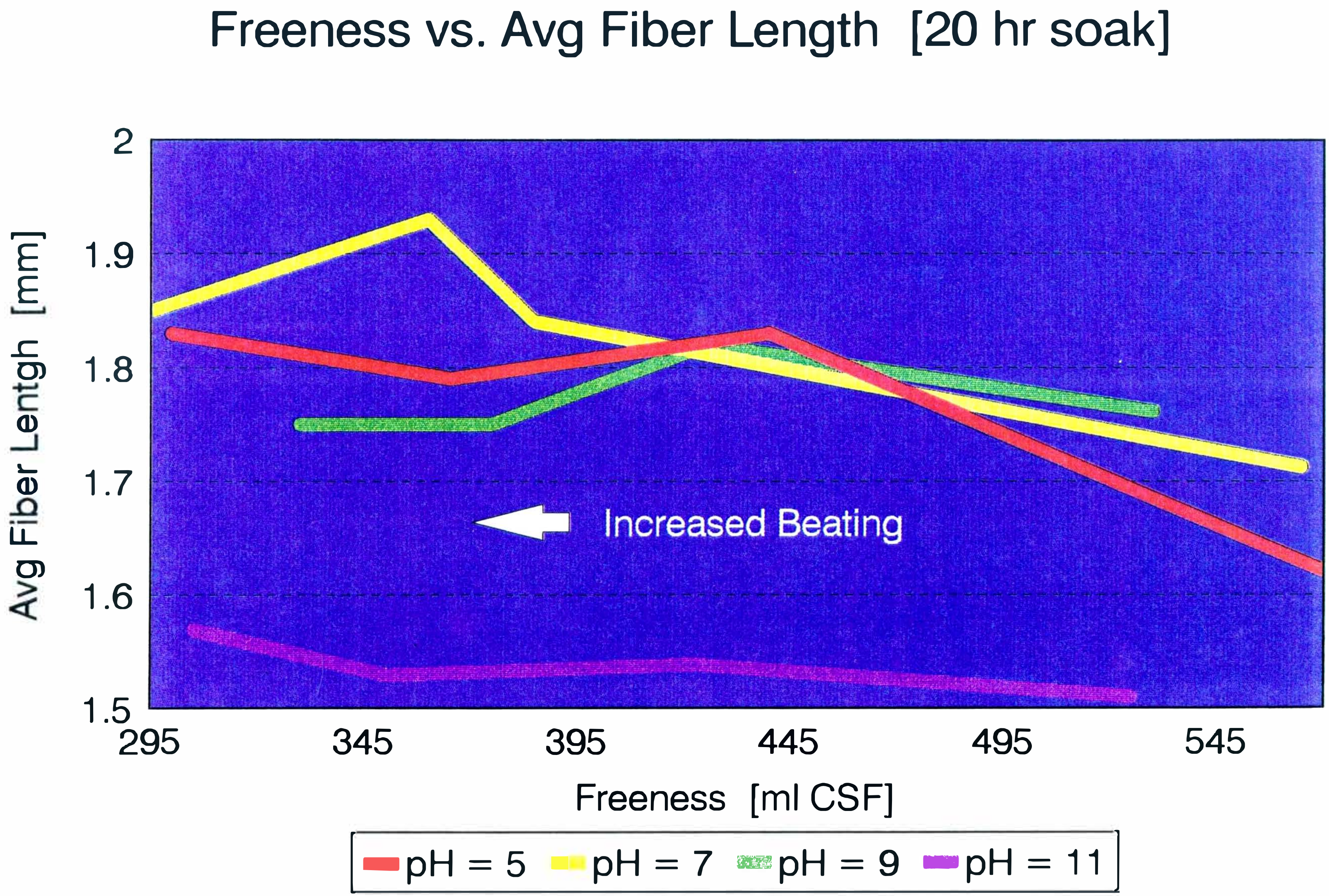
Graph 16



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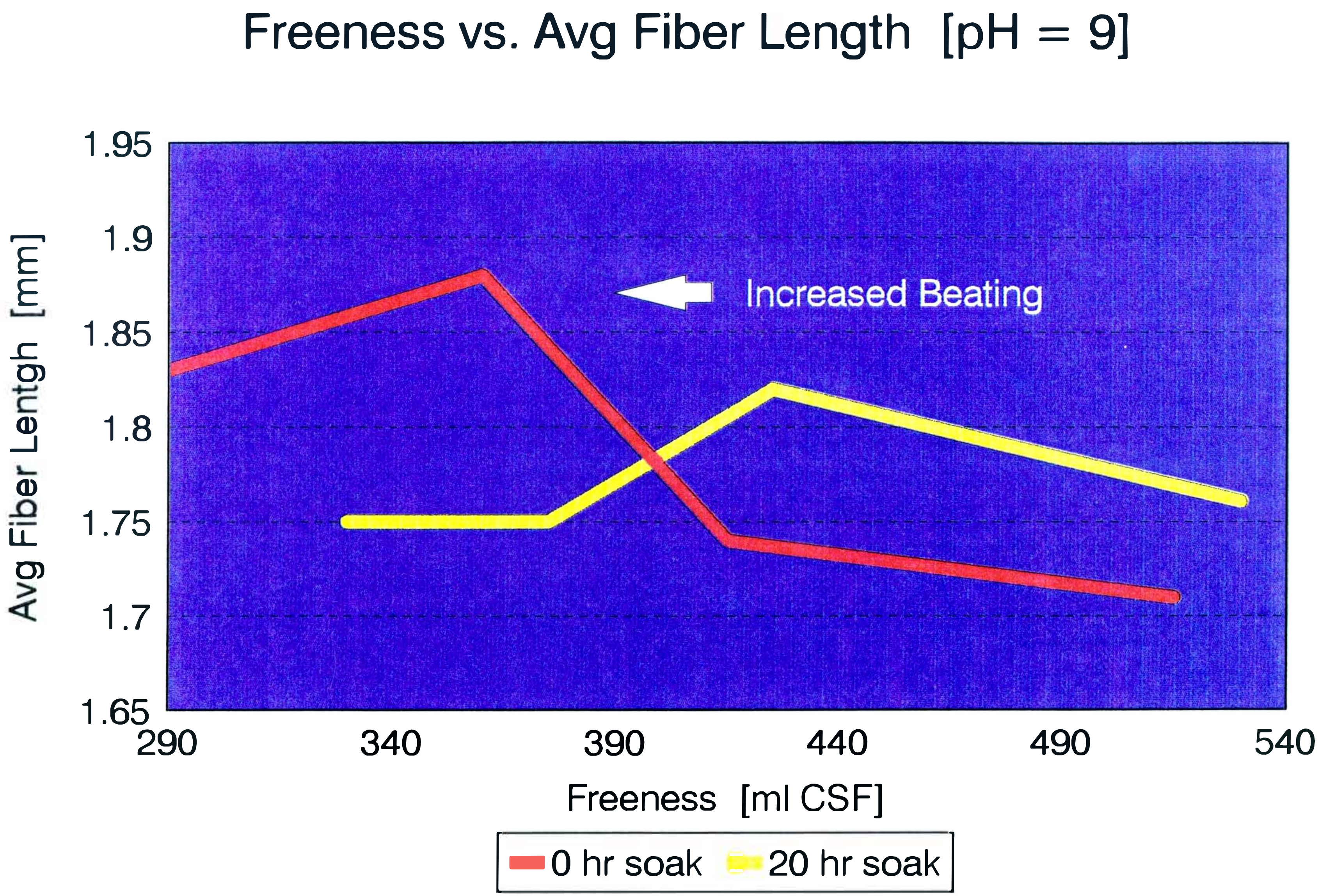
Graph 17



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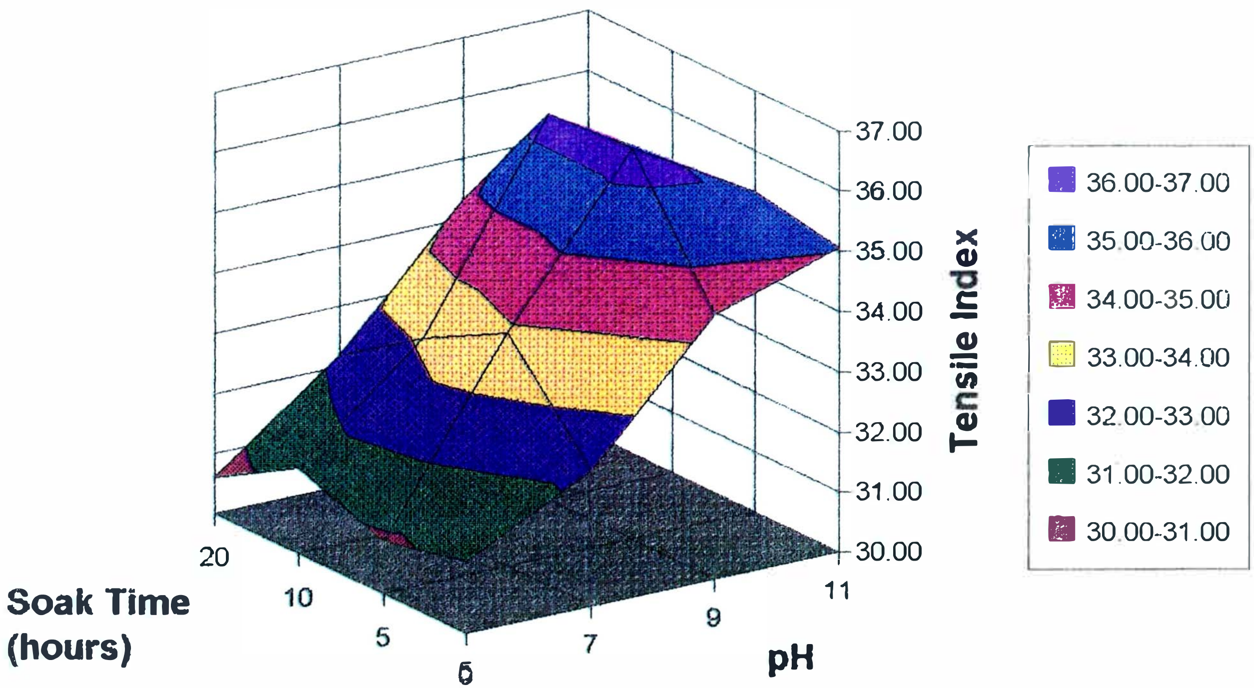
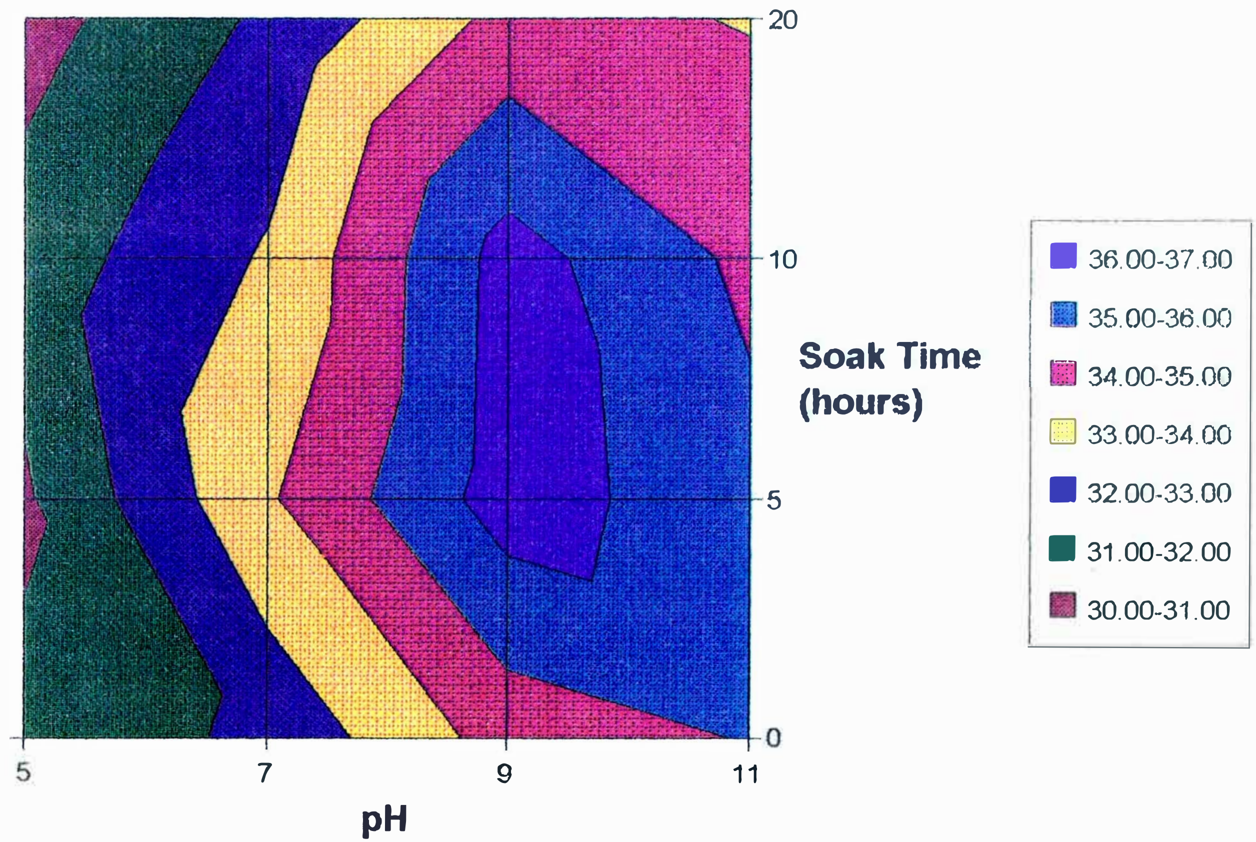
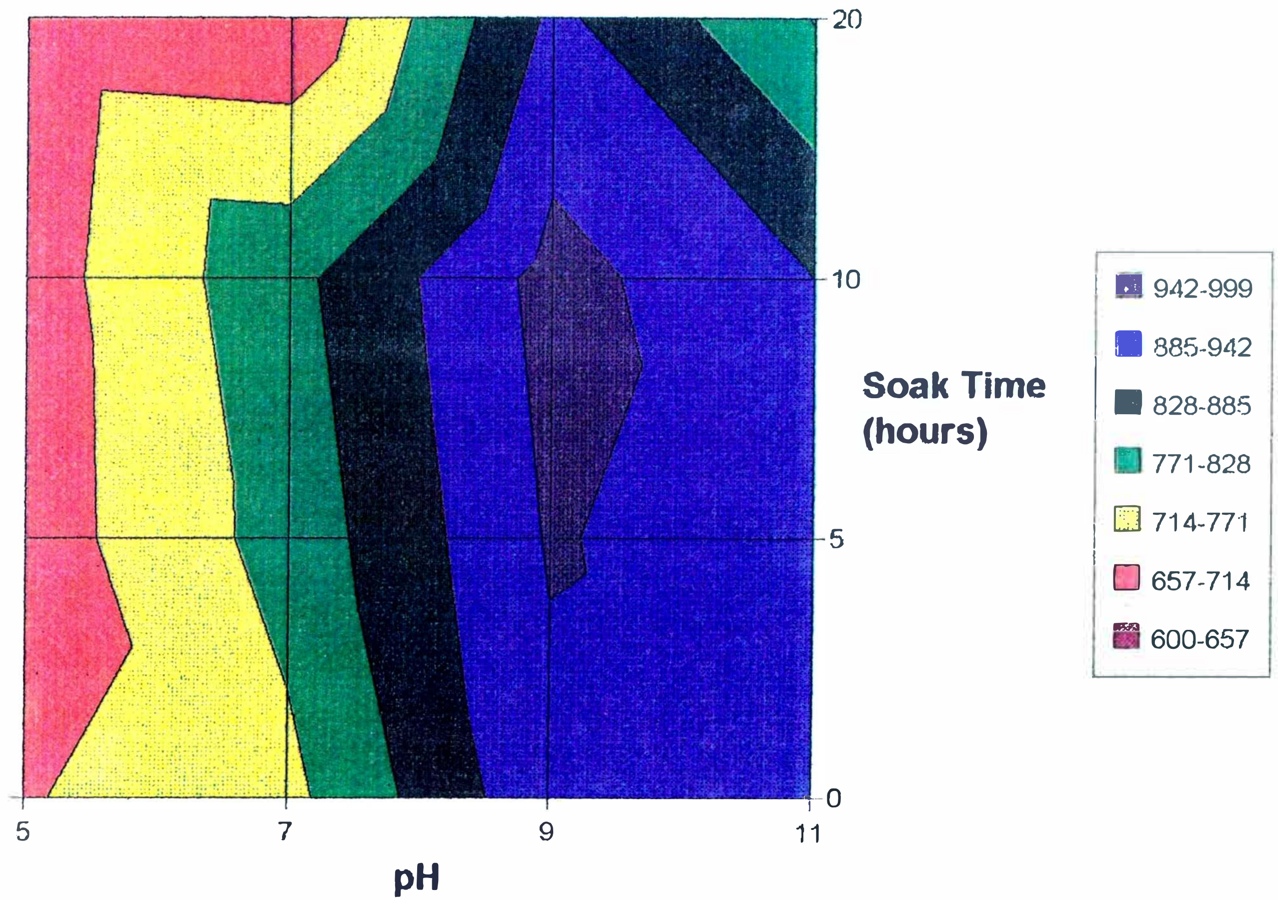
pH and Soak Time vs. Tensile Index (side view)



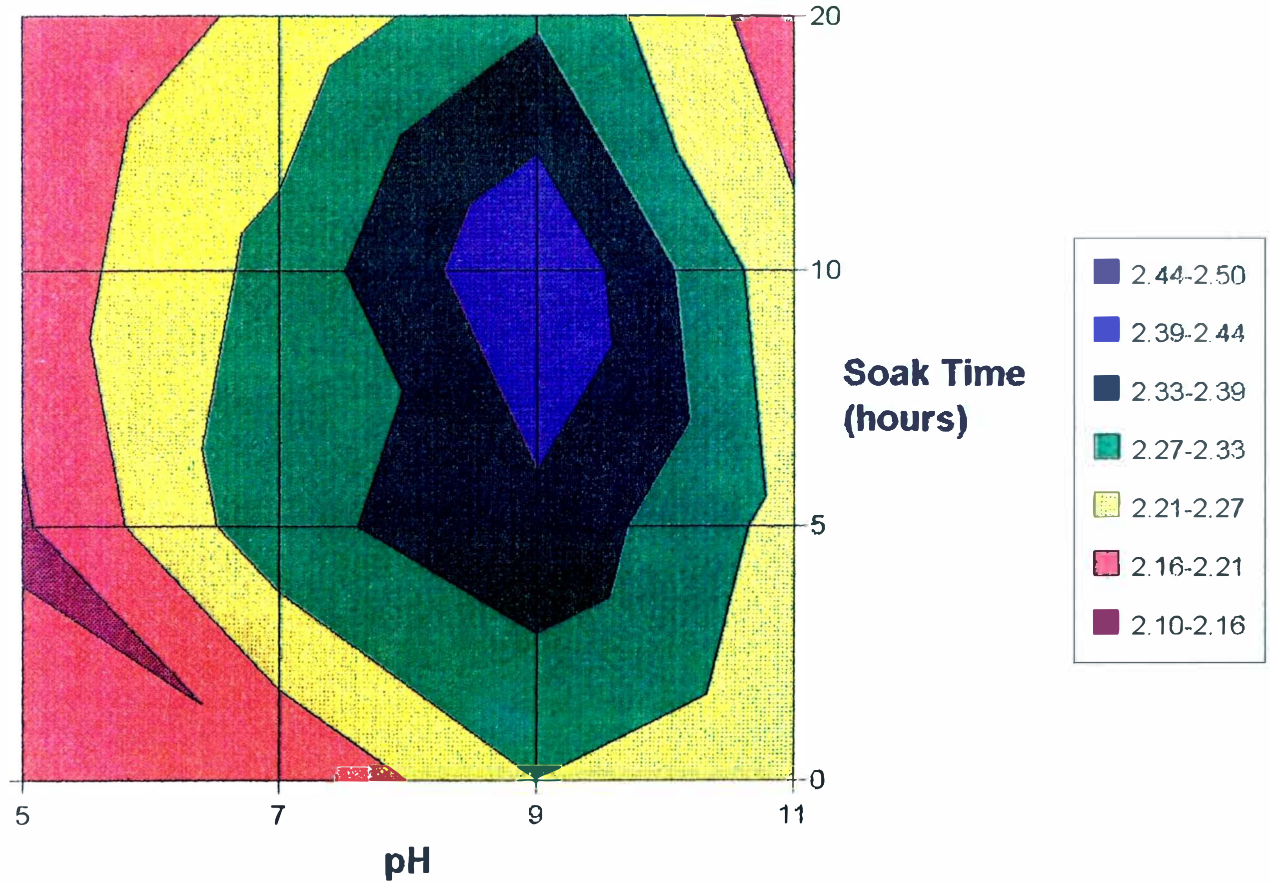
pH and Soak Time vs. Tensile Index



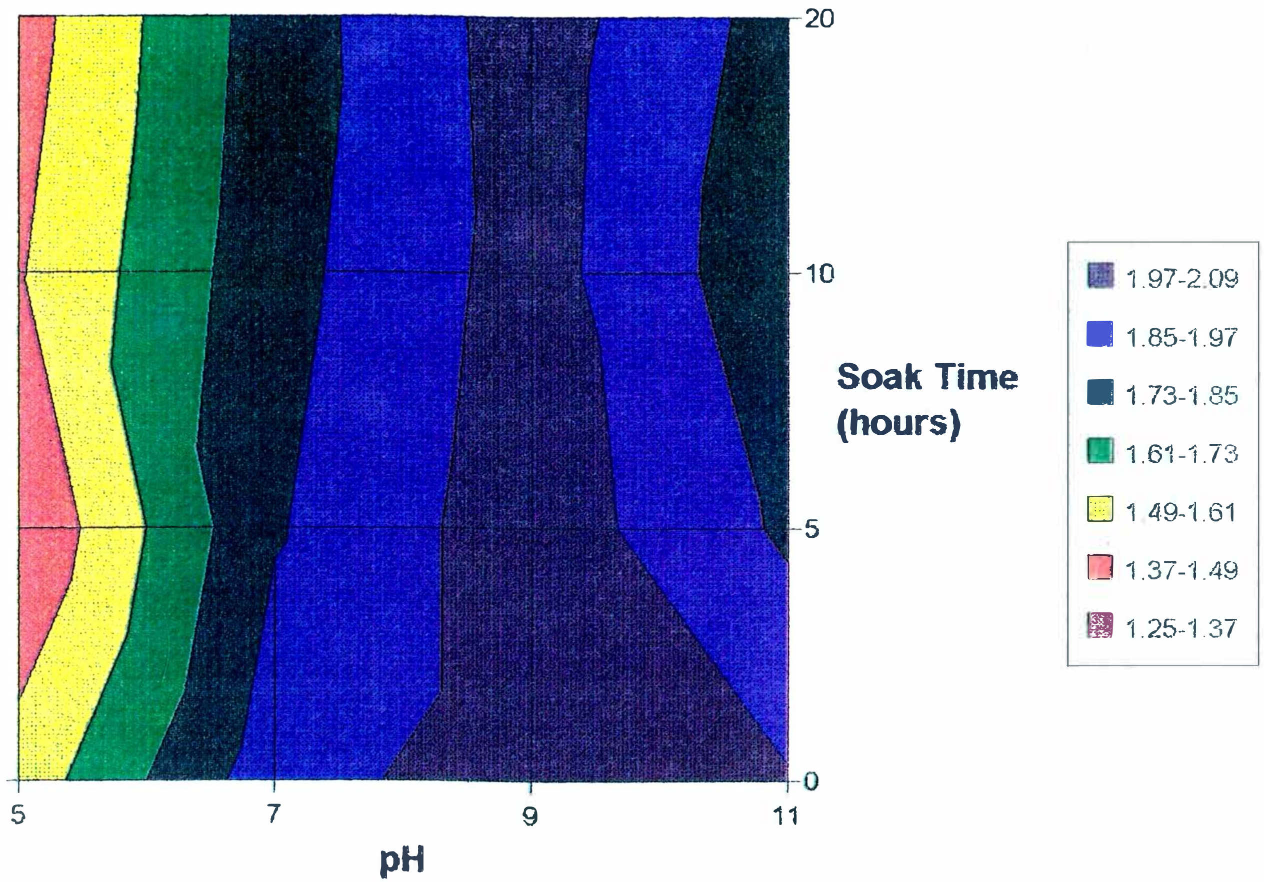
pH and Soak Time vs. TEA Index



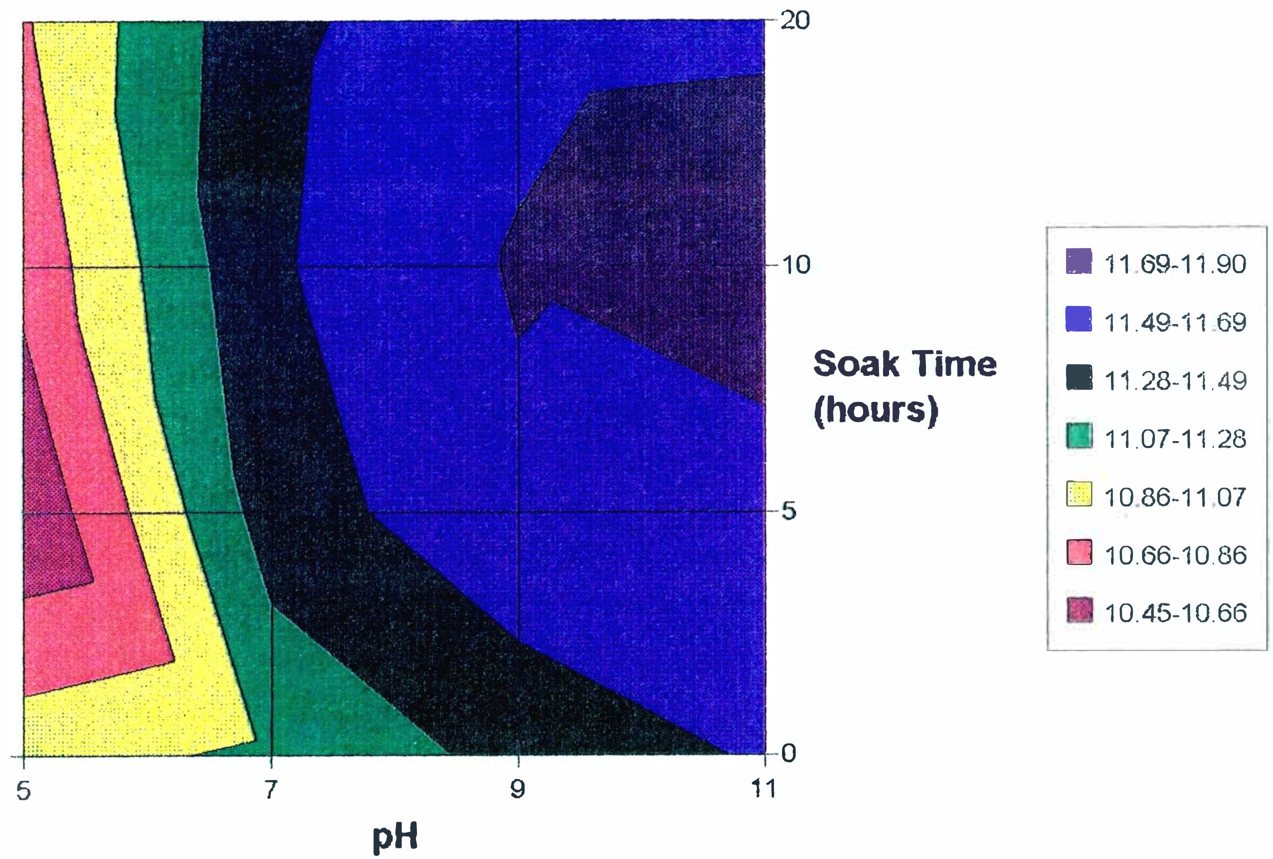
pH and Soak Time vs. % Stretch



pH and Soak Time vs. Burst Index



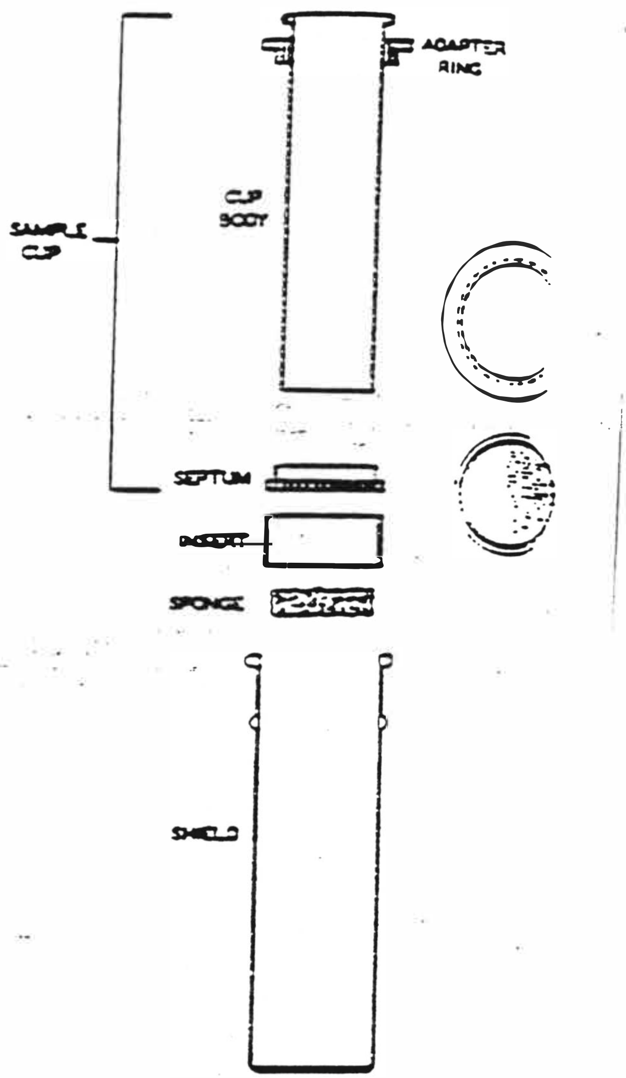
pH and Soak Time vs. Zero-Span Tensile Strength



APPENDIX THREE

APPENDIX III

Centrifugal Cup Apparatus for Water Retention Value



CENTRIFUGE CUP ASSEMBLY