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Investigation of Effects of Ph, Ionic Strength, and Drying Temp. on Dimensional Stability of Film

by

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Submitted in Partial Fulfillment of the Requirmemts for the Degree Bachelor of Ssience

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1964

SENIOR RESEARCH THESIS

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MAY 1, 1964

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H. N. Parsonage G. A. Pittman \Box An Investigation Into The Mi

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pH, Ionic Strength, and Drying The prombers

On the Dimensylonal Stability of a Fine

Grain Priuting Film

 $\mathbb{M}(\mathbf{x}) \leq \mathbb{C}(\mathbf{x} - \mathbf{1}) \quad \text{and} \quad \mathbb{R}$

Harry N. Parsonage

George A. Pittman

 $\label{eq:2} \frac{1}{2} \int_{\mathbb{R}^3} \frac{1}{\sqrt{2}} \, \mathrm{d} \mathcal{L} \,$

 $\frac{1}{\sqrt{2\pi}}\frac{1}{\sqrt{2\pi}}\left(\frac{1}{2}\right)^{\frac{1}{2}}\left(\frac{1}{2}\right)^{\frac{1}{2}}\left(\frac{1}{2}\right)^{\frac{1}{2}}$

Dedicated To:

Our loyal, devoted and understand Wives - JoAnn and Judy

ABSTRACT

An investigation into the effects of ei-ruleal chases made in a processing system and drying Louperature wit. , ,, ,, , to dimensional displacements of a final image was curried out using * Eastman Kodak's # 8U30 aerial duplies ing film. Image di--r,i: icnts over both long and short distances were measured.

Over approximately 162 mm., drying at a temperature equal to that of processing, 70° F, resulted in no significant spatial d spacement of the final image, while drying at higher temperatures produced correspondingly larger displacements. A pH system between bathe similar to that found in a conventional process resulted in dimensional displacements, over the short distance or approximately ⁹⁰ microns, less that processing systems employing gradually increasing or equal pH's throughout the baths. The salt concentrations investigated produced no significant effect on dimensional displacements.

An Investigation $I_D(x)$ The $V_L(x)$

pH. Ionic Strength, And Dry (v. Verschaft) On The Dimensional Stability, Of A Fina Grain Printing Emuls on

Previous investigations of dimensional Claudace on: the photographic image have been more concerned with μ apping the actual displacements under a given set of processing, drying and/or atmospheric conditions. Conceivably previous workers had hoped to map the distortions to be expected so that the effects due to these factors could be removed from data obtained from films and plates. It was the intention of the authors to investigate the effects of quantitative changes of the process on the dimensional dispiacements of a photographic image. Displacements of the photographic image appear to be due primarily to the expansion and contraction of the \pm gelatin and the support.

To investigate the effects of such changes three factors were chosen that have a direct bearing on swelling of gelatin and on expansion and contraction of the base. The three factors Investigated were:

. 1. Changes in pH between processing solutions.

2. Changes in salt concentration between processing solutions.

3. Drying temperature. \.^

Changes in ph --- L . As fifa is pvt in or aje. the gelatin expands. When the film goes , som this all the to the acidic stop bath it goes through a region of for the gelatin, the isoelectric point, and then swell. reaching the low pH of that solution. Fixation and washing \cdots and change the swelling of the gelatin depending on the haraon. pH of the fixing bath. Hence, changes in ph durium, the plate of : process cause stresses on the film through successive extremi and contractions of the gelatin. (See Diagram $\#$ 1) \bullet

Changes in salt concentration- $-\frac{2}{\pi}$ Swelling of gelatin is usually lowered by increasing the salt concentration of the solution. ^A developer, of somewhat high salt concentration, is followed by a dilute stop bath, which in turn is Tollowed by a fixer of higher salt concentration than that of the developer. Tap water used for washing has no salts of any consequence. Therefore, as with pH changes within a given system, the salt concentration changes between solutions in the photographic process cause successive expansions and contractions of the gelatin. These may be contributing factors to the dimensional displacements of a photographic image. (See Diagram $#2)$

Drying temperature $---$ ³ An increase in temperature generally causes film to expand. But, if the increase in temperature is not accompanied by an increase in absolute humidity, the relative humidity will decrease. The film will contract with a corresponding decrease in relative humidity (RH). During the drying process the film is subjected to both high temperatures and high RH at the beginning of drying, causing expansion. As drying continues and the film dries the RH decreases causing the film to contract. The drying process imparts

 $\frac{1}{2}$

both reversible and irreversible changes on . he as a \mathbb{R}^d The Reversible, or temporary, changes occur due to a second of the film according to the temperature coefficient -nike the irreversible changes are due to the shrinkage consed by loss of solvents from the film base. Here again there are opposing a within the film that could lead to dimensional diaments. All the final image.

Printing System

The film samples were printed with ultra violet radiation, o $3400-3800$ A, in contact with a master glass plate, The master was a spectroscopic plate with an image of fine ruled lines (similar to graph paper). The lines were transparent, approximately 0.1 mm wide, on a dense background and formed squares four milimeters on a side. The master glass plate was $9\frac{1}{4}$ X 1 inches. Five inches of the center was transparent. In this transparent area the stop tablet was placed. (See Fig. 1) The master and step tablet were placed in the platen of a Pittman Model B Control Sensitometer (xenon flash) $\frac{1}{T}$ The sensitometer was modified to print the step tablet by light and the reseau grid pattern by ultra violet radiation, as isolated by using the Wratten 18A filter. The ultra violet was used to print the grid in order to print as sharp a line as possible with this printing system. The light exposed step tablet was used to check sensitometric control of the process. Exposures were made in contact under a pressure of three pounds on the platen.

3.

 $, 1$

Processing Conditions

pH Systems --- Two systems of graviorally disc between baths and on of no changes in gill shown bathe to a system of pH changes between baths condlar to that we wil process, where the changes are very large. (Let \mathbb{R}^n be a set of \mathbb{R}^n ,

Table 1

pH systems investigated

*Normal pH values obtained from monsurements of freshly prepared solutions of Kodak's formulas: D-76, SB-1, & F-5.

Salt Concentrations Systems --- Salt concentrations were expressed as ionic strength in order to take into account the effects of the charges on the ions. The relationship used for the estimate of ionic strength was:

$$
I = 1/2 \sum (m_1 \cdot z_1^2)
$$

where I represents ionic strength and m is the concentration, expressed as molality, of each ion with a charge of z_1 . (See appendix for sample calculations) Since the film must start at a high ionic strength, I,

in the developer and go to the wish of $I \subseteq \emptyset$. strengths must necessarily start at a ; and with Three systems of ionic strengths were formulated ard fourth system representing the normal or conventional formulated systems consisted of two rates of gradual ϵ between baths and a third system of ionic strengths equal to . the developer for all baths except for the wash which was zero. (Fee table 2 for values) (Diagram $\frac{1}{2}$ 2)

Table 2

Ionic strength systems investigated

*Normal ionic strength values calculated from Kodak's formulas: D-76, SB-1, & F-5.

Drying Temperature --- The temperature equal to that of processing and two higher temperatures were compared.

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Film Handling

Eastman Kodak's 8430 aerial dupleating, 70 as an tri-acetate base film was used in the investigation. removed from the spool and pre-conditioned to 50 $^{\circ}$ twenty-four hours prior to exposure. All of the fileexperiment was exposed at $47 \stackrel{1}{\sim} 1\%$ RH at $\frac{1}{2}$, $\stackrel{1}{\sim} 1\%$ using the set previously described at 20 second recycle times.

Processing solutions, including the wash and wetting agent, were held at $21 \stackrel{+}{\sim} 1^0$ C. The relative hunderity of the laboratory during processing was $53 \pm 3\%$ RH. The exposed strips were placed on a process rack to enable three different treatment combinations to be run at once? Each strip was processed in a freshly prepared liter of developer, stop bath, and fixer. The samples remained on the rack for processing and washing. After washing, the samples were bathed in a $2\frac{1}{4}$ wetting agent before being squeeged. The squeeging process was done using a roller driven film squeege machine employing two viscose sponges inclined 30° to the film. Each film sample was dried horizontally in the center of a shelf of the 7 Pittman & Parsonage Control Strip Dryer. The dryer had capabilities of $\pm 1/2$ C temperature control within the ranges used.

Processing Solutions

Developer --- A ferrous ethylenediaminetetraacetic acid ($Fe₂EDTA$) 8 developer similar to that formulated by James was chosen because of its low pH dependency with respect to its activity. At the concentration

o.

used the developer produced approximately confivation charactoristics at pli values boi seen 4.00 and 10.00.* developer activity is primarily pH α dependent the derived is not. As pH increases the pK increases thus increasing $\overline{\cdot}$ ionic strength of the developer. $9 - 6$ odium sulfate was and $1 + 6$ a constant ionic strength of 2.2' with changes in devalues \ldots . constant appendix for sample calculations) Since sodium ions were present from the chelating salt Na_kEDTA and sulfaue ions, were present from the $F \circ 30_{ki}$. * the use of Na_2SO_4 to balance ionic strength added no new constituents to C the developer. The pH of the developer was adjusted to the desired values using glacial acetic acid. (See Table 4 for formulation at various pH values)

Stop Bath $---$ Since the developer is pH independent with respect to activity the use of an acid stop as in the conventional process could not be done. The technique of a bromide stop bath as used in processing nuclear emulsions was used. A bromide bath of 23 $1/2$ g/1 KB r stopped development as well as a conventional stop bath when using the Kodak formula D-76 developer. The pH of the stop baths were maintained with low ionic strength buffers. 12 Again sodium sulfate was used to adjust the total ionic strength of the solution as required.

Fixer --- A fixer that functioned in both acidic and basic states with unusually low ionic strength was formulated using ammonium 13 thiosulfate as the silver halide solvent. Sodium formaldehyde bisulfite * See Figure $\#$ $\frac{1}{2}$ guardian set $\frac{1}{2}$ guardian

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$\mathcal{A}(\mathcal{A},\mathcal{O})$.

as a cavelopers rows situated with 98.9% acetic acid.

in low concentrations provided the necessary formation for the pH's above and below 7.00. The total iou o strength ϕ adjusted as required usIn(5 codilim r.vilf'uto. '\.'*>.'ionie . , . :.:,_>. similar to those used in the stop bath were used to maintain an

Method of Measurement.

The glass master and film samples were stored at 501% at at \circ 1912° C for eight days prior to transportation to Data Corporation for measurement. Long distance measurements were made on the Man ;- Data Micro-Analyzer* which was used as a micro-comparator. $^{1/4}$ The Whitfield room, in which the microcomparator was located, was held at 50.11/ MI - 92 and $23.91.5^{\circ}$ C for the three days of measurement. The instrument was calibrated with a glass step tablet (for measurement of the glass master). and with a film step tablet (for measurement of film samples) to account for the different bases of the specimens. Focusing of the instrument was done electronically using the maxium fluctuations of the recording volt meter. This was the best estimate of a sharp focus for the granularity would be at Its greatest value.

The samples were placed on the scanning stage and aligned with the ^X axis of the stage using the edge of the film. Further alignment with the ^X axis was accomplished by pivoting the stage in the ^Y direction until the line being measured was perpendicular to the ^X axis. In order to start at the same position on the line the sample was moved in the ^Y direction a constant distance (.771 mm) down from the horizontal line that Is common to both sides of the master. The stage *See Figure #10

was moved manually in the X direction until the dense, of the vertical line was 1.0 as indicated by the reonirm . . . A second measurement was made on the same line $1/42$ measurement was previous measuring point. The screw jositions for these ' recorded. Corresponding measurments were mode ona s.jak. line at the other end of the film suaple. In all cases the some manner were made on the same side of the vertical lines of all the sumples. (See fig. 2) Making measurements from the corresponding sides of the lines reduced the error due line width expansion end/or contractions,

Exact alingment of the samples with the direction of measurement was impossible. Therefore, not only would the distance between the two lines be incorrect due to the slight angle of the sean but the chance of measuring to imperfections on the line was increased. In order to compensate for the slight angle of scanning the second line was measured as was the first. The distance In the ^Y direction that the stage had to be moved to bring the measuring point the same distance below the horizontal reference line (0.771 and 2.313 mm) as for the first line was recorded. The displacement in the ^Y direction was an estimate of nonalignment and rotation in the sample. (See fig. $3)$

Screw position data and displacement in the ^Y direction due to non-alignment were punched on cards and by the use of the IEM 1620 computer, the actual distances were calculated.¹⁷ The primary mechanism of the program was the use of the Pythagorean Theorem. The actual distance was the hypotenuse of a right triangle in which one leg was the displacement due to rotation and the other leg was the distance measured. 'H

y.

Microdensitometer. Because the lire week 0.1 mm there was no problem of edge atignwent. The serve were measured at a density of 0.8 . The time position to : was located at a constant distance down from the hor: with the long measurements. (This distance was ω is the 0.771 mm and was visually placed) Similar to the long results of α the screw was turned manually moving. Her sample along until the recording volt meter indicated a density of 0.8 . The serve a sition was recorded and the stage backed off. The 'less we' oa forward after taking up any slack in the screw the cade of recorder indicated that the other side of the line had been reached. The stage was moved until the recorder came down to a density of 0.8 ; then the'screw position was recorded. Duplicate readings were made on line widths.

The glass master was measured on both instruments. Using the micro-comparator the distance between the two lines chosen was measured (long measurements) at an edge density of 1.0. The Ansco Microdensitometer was used to measure the line width using the 18A filter over the photocell. Focusing was done. with maximum fluctuations of the recording volt meter. Several traces were made using different degrees of knife edge focus to find the best estimate of the original line width. Thus, the line ~ $\mathcal{L}^{\text{max}}_{\text{max}}$ was measured in a similar manner as this line printed the samples, both with the same UV radiation and both with diffuse illumination on the master.

Line widths were measured usin

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Sensitometric Adjustments Of Line Widthe

The processing systems were not exactly early ψ sensitometric characteristics that they produced. Any disc that would be found in the line width when one of the same of constituents were altered could be caused from either:

- 1. the chemical change. ω_0 is we see as
- 2. the slight sensitometric differences that v_{obs} a result of these chemical modifications.

'An analysis of the line width measurements indicated that, $x \in \mathbb{R}$, alpha risk of 0.001, there was a significant difference in lime widths due to the pH systems used. However, associated with each of these pH systems there was a slight sensitometric difference (Sec fig. 4).

The sensitometric difference could have caused a larger line width to occur than if the pH alone was changed (with no accompanying sensitometric shift). There may have been no line width change at all. The solution to this problem lies in an analysis of the components that contributed to the line width difference and then factoring out that which can be attributed to the sensitometry.

All of the samples were measured at a constant density of 0.8. Since the shape of the characteristic curve is different for each sample, each of these 0.8 densities was printed by a different Log E. (See fig. 5) Because the edges of the line on the master had some finite slope, the edges acted as a continuous wedge modulating the exposure to the edge of the sample. The Log ^E produced from the edges of the master's line was also finite. The Log ^E difference required to produce a constant density on the samples with different characteristic curve shapes is

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equal to the density difference of a second of the second Therefore, the line width of one sample was rot needed. the same distance on the master as another sample. (he. \mathbb{R}^3 a. The solution to the problem, then, is to go to the original sensitivity associated with each sample and find what the distance was on the set that printed each sample. Subtracting the samples dicirese from an distance that printed that particular sample would then give the deviation in line width due only to the pH uhanges made, having eliminated the effects due to sensitometry entirely. 16

With a 99.97 confidence there we ائی در دیا
مکانستان ما wewith a war alffar the in line vidth ius to the drying temperatures used. 27 Just as with the pH systems used, there was a significant difference in speed and slope with respect to the different drying temperatures. Here again, there is the problem of which caused the change in line width:

- 1. the different drying temperatures, or
- 2. the different density levels caused by the changing of drying temperature.

When the- corrections were made for the sensitometric differences from the pH systems, the corrections for sensitometric differences due to the drying temperatures was also done. It would be impossible to factor out just the effects of pH since each sample had some drying temperature. Fig. 8 indicates the relation of drying temperature to line width before (with falsified distances due to sensitometric $\mathbb{Q}^*_{\leq 0}$ differences),' and after with the sensitometric differences with each temperature factored out.

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Discussion of Results

Long measurements, 162 mm --- Since there was a in temperature of 3.9°C from the time of exposure to measure film and glass master both expanded. The glass master expanded while the film samples expanded 29. according to their and ω temperature coefficients. 24 was subtracted from the file and same measurements for the final averages of the long measurements.

Line widths, O.1 mm --- Recense of mechanical manufactured error in the microdensitometer the measured cage of a line would be at a lower slope than that of the actual edge. An error of only call micron would be multiplied by four because of the system being used to obtain a final measurement of the difference in sample line width and master line width. A factor of two from the two samples being compared. and a factor of two because there are two sides to each sample. (See fig. 9)

Therefore, the values given for the deviation from the master over the line widths are incorrect. But, the relation between the numbers are the same. In this case, the largest negative number (-5.1) is the 'best' because it is the smallest deviation.

Arithmetic speed --- The arithetic speed system was based on the log exposure required to produce a density that is 0.3 above base plus fog. 18 This corresponds to a density just into the straight line of the characteristic curve.

Average Gradient ---The maximum gradient obtained over a 0.6 log exposure range as found by the IBM 1620 computer with H.N. Parsonage program $#3.$ ¹⁹

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Conclusions

Long measurements, 162 am --- The a sense significant difference was found over the long measure of the pH and ionic strength systems investigated.

With an alpha risk of 0.01, there was found a significant difference in the long dimensions with respect to drying thermarkure. The 70[°]F temperature produced an image with dimensions closest to the original dimensions of the master. The two other tempormulars resulted in film with dimensions significantly less than that of the masser.

Line widths, 0.1 mm $---$ With a bets risk of 0.05 , no significant difference was found in line widths with respect to any of the ionic strength or drying temperatures used. Although there was a significant difference in line width with a change in the drying temperature, it was not the heat itself that caused the change, but the sensitometric differences. that resulted from those temperature differences. With no sensitometric differences, there would be no significant difference in line width at any of the temperatures tested.

With an alpha of 0.05, there was found a significant difference in line width with respect to the pH systems used. The Normal and Gradually decreasing pH systems produced lines significantly smaller than the other two systems.

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FOOTNOTES:

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- $\begin{split} \frac{\partial}{\partial t} & = \frac{\partial}{\partial t} \frac{\partial}{\partial t} \frac{\partial}{\partial t} \quad \text{if} \quad \\ & = \frac{1}{\partial t} \frac{\partial}{\partial t} \frac{\partial}{\partial t} \end{split}$ 6r. Parsonage, H.N. and PIttman, G.A. 'Programs for IBM 1620 Computer' May 1964 Rochester Institute of Technology Rochester, New York

Figure $# 2$

 \overline{a}

The long distances on the samples were measured from the right side of line A to the right side of Mine Bo Thus, if the lines expand errors in the long measurements would be at a minimum because the sides would be expanding in the same direction - at the same time.

Figure $#$ 5&4

Sample Calculations of Ionic Strength

Ionic Strength represented by the sysmbol I follows the equation:

$$
\mathbf{I} = 1/2 \Sigma \, \mathbf{M}_1 \mathbf{Z}_1^2
$$

M_i represents the concentration in Molality and where Z_i represents the charge on the ion.

 $I = 1.120$ For

Buffer I = 0.04

 \therefore 1/Mw/ 23,5g/1 = 23.5/114.02 = 0.196 M **KBr** I

 $50\frac{1}{2}$ Ma $1/2((2 \cdot X \cdot 1) + (1 \cdot X \cdot 2))$ $0.884 =$ $= 1/2(6\lambda)$ $3X$ 3 $= 0.295 M$ $= 142.05$ \mathbf{x} 42 grams $Na₂SC$ \approx Mw of $N_{\mathbf{a}_2}$ SO_L

Hence the addition of 42 grams of sodium sulfate to the standard amount of bromidea and buffer should give the desired ioni strength.

Sample Calculation of Ionization of ETDA Hon

 $FeSO_{\frac{1}{4}}$ 20 g/l / 152 g/mole = 0.131 Molar \texttt{Na}_2 EDTA 60 g/l / 380.2 g/mole = 0.157 Molar

 $\hat{\mu}$ O ml. of $H^{\circ}C_2H_3O_2$ required to obtain pH of μ . O $I = 0.680$ $1.209 + 0.680 = 1.889$ Desired I = 2.257 $-$ 1.889 0.368 / $3.0^* = 0.123$ 0.368

 0.123 X 14 α g/mole = 27.5 grams of Na₂SO₁ to be added $2 - 4$

*

See sample calculations of ionic strength for details.

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Since the samples were exposed with ultra violet radiation and the step tablet was exposed by light a relation between these two methods had to be determined. The transfer function, in quadrant $#$ 2 was used to determine effective log UV that the samples recieved, using the whitel light control strip from each sample. From this effective exposure differences, the different distanced on the master were calculated for each sample.

In order to have a direct comparison between response variables of different and indirectly related units the above graph was prepared. Each of the response variables. speed, and dimensional differences, are plotted with their averages normalized to \overline{X} . The scales of each of these variables are plotted with the standard deviations for each at unit length. Therefore any changes in the value of unrelated variables can be compared through changes of eachs relative change from the one standard deviation.

MANN-DATA MICRO-ANALYZER

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alue To any one Portmany

"a pit system - " - poor wording

I still don't like the wording of the "displacements over troll, were and also it distances" Seich does not tell what it is about

Kodale Verdale desolution sinte (1900 plates include many enversions) 15 No West control Att 22 Whowever In Rtl volus measured ? all exited dryen? It would probably be better to take RH and Tony, at intake arrivantate RH at dryer temperature, which gives the state of the our real hing the films; "The exit, lite indicate how much was Turn up.

p14 The largest negative number with " because it is the smallest ??? How can you say which ? There probably was me spread of wage the platty replace mage over the mage falling on teapion, so to at a final difference was multer of the officing factors