THE INFLUENCE OF AGED DISTILLED WATER ON PULP PAD PERMEABILITY[1]

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ABSTRACT

Past experience had shown that pulp pads permeated with aged distilled water always experienced a permeability decrease which was independent of the dissolved gas content. This loss could be regained, at least in part, by subsequent permeation with freshly distilled or filtered aged distilled water. The size of the blocking agent in aged distilled water was found to be between 1.2 and 5.0 microns, which falls within the size range determined by other workers for stable bubble nuclei in tap water. A dilute particle suspension exhibited a behavior identical to that of aged water, but microscopic analyses of filters showed no difference in the number of contaminants in freshly distilled or aged distilled water. Boiling (100 C) greatly reduced the deleterious effect of aged water. Although aged distilled water decreased pulp pad permeability, the specific filtration resistance of pulp slurries was not increased by aged distilled water and tap water when compared to freshly distilled water and filtered tap water. The presence of bubble nuclei would explain the effect of aged distilled water on pulp pad permeability, but how they are stabilized and why they develop is not known.

INTRODUCTION

In a previous publication (Gertjejansen and Hossfeld 1967), it was shown that when wood pulp pads were permeated by distilled water, constant permeabilities were readily obtained from the very onset of permeation (Fig. 1), provided that the pulp slurry was bubble free prior to pad formation and the permeating water was either freshly distilled or properly filtered if it was aged.

Decreasing pulp pad permeabilities have been observed by several investigators (Anderson, Gortner, and Schmitz 1941; Carroll and Mason 1952; Goring and Mason 1950; Robertson and Mason 1949) and various explanations have been put forth for this phenomenon, most of which relate the decrease to the pad itself and not to the permeating liquid. However, Gertjejansen and Hossfeld (1967) showed that a decrease in pulp pad permeability was independent of the pulp pad and solely a function of the permeating water. Specifically, it was a function of the age of the distilled water and was independent of its dissolved air content, indicating that gas was not evolved during permeation. It was also found that permeability lost to aged distilled water could be recovered, at least in part, by subsequent permeation with freshly distilled water or properly filtered (0.22 micrometer pore diameter Millipore filter) aged distilled water (Figs. 2 and 3). It was hypothesized that the deleterious effect of aged distilled water on pulp pad permeability was due to bubble nuclei in the aged water which had grown and stabilized over time and were functional in plugging pad pores and hence decreasing permeability.

The present study was initiated to define better the properties of aged distilled water and to determine further its importance to pulp pad permeability.

THE CONCEPT OF THE BUBBLE NUCLEUS

The hydrodynamicist, acoustical scientist, and hydraulics engineer explain the cavitation of liquids at relatively low negative pressures by the presence of stable bubble nuclei in the liquids. The theoretical tensile or cohesive strength of water has been estimated to be between −500 and −1,000 atmospheres, yet these values have never been attained in practice. This discrepancy has been attributed to the presence of persistent, stable, minute bubble nuclei which create "weak spots" in water and allow cavitation and boiling at low pressures.

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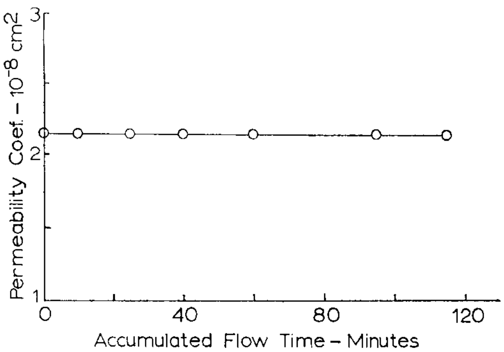

FIG. 1. Constant permeability of an unbleached semichemical aspen pulp pad using unfiltered freshly distilled water at 97% dissolved air content.

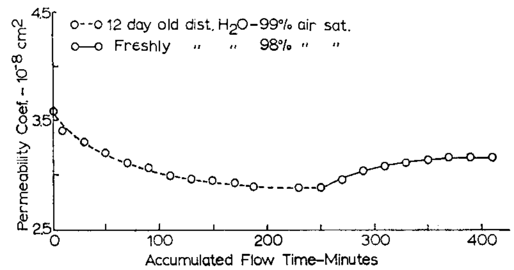

FIG. 2. The effect of aged and freshly distilled water on the permeability of an unbleached semichemical aspen pulp pad.

Since the pioneering work of Harvey et al. (Harvey, Barnes, McElroy, Whiteley, Pease, and Cooper 1944; Harvey, Whiteley, McElroy, Pease, and Barnes 1944), a multitude of investigations, primarily with water, have been conducted which deal directly or indirectly with the concept of bubble nuclei. A portion of these have been cited in this paper. It has been almost universally concluded that bubble nuclei do in fact exist, but their existence is a paradox. Theory predicts that gas bubbles in undersaturated solutions will dissolve in finite time (Epstein and Plesset 1950; Keller 1964; Plesset 1964). In saturated and supersaturated solutions, the same can also be true, because of surface tension, provided that the bubble radius is not too large (Keller 1964). Therefore, if minute bubble nuclei do persist in water, some sort of stabilization mechanism must be operable, and several theories have been put forth to describe this mechanism. Harvey et al. (1944) and Liebermann (1967) have postulated that bubble nuclei stabilize themselves in cracks and crevices of hydrophobic contaminants which are present in water. Because of the wetting angle of water on the contaminant and a subsequent reversal of surface tension, a minute bubble associated with the contaminant can be in stable equilibrium with the gas tension of the solution. Fox and Herzfeld (1954) attributed the stabilization of a nucleus to an organic film around the minute bubble which acts as an elastic shell and a barrier to diffusion. Strasberg (1959), in a rather extensive study, could not distinguish between these two theorized types of nuclei. However, he did conclude from his experimental results that nuclei were not solid particles only and that they did contain undissolved air in some sort of equilibrium with the air dissolved in water. Turner (1961) theorized from his investigations that nuclei are stabilized by a "compressed wall" formed from unavoidable contaminants in water. Messino, Sette, and Wanderlingh (1967) support this point of view because of their own experiments. Thus three theories exist, but conclusive evidence is lacking in support of one or more.

Several methods have been employed to eliminate the effect of bubble nuclei: pressurization, filtering, centrifuging, degassing at room temperature under vacuum with sound or mechanical energy, and degassing by boiling at 100 C. Harvey et al. (1944;

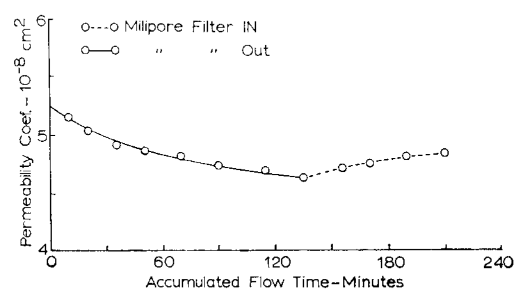

FIG. 3. The effect of unfiltered and filtered (0.22 micron Millipore filter) deaerated aged distilled water on the permeability of a bleached southern pine kraft pulp pad.

1945; 1947) showed that water which was subjected to pressures in excess of 1,000 atmospheres could be heated to 202 C before bursting into vapor, the function of the pressure being to force the nuclei into solution. Strasberg (1959) using pressures up to 6 atmospheres, and Iyengar and Richardson (1958) using up to 20 atmospheres, showed that sound energy required for cavitation increased as static pressure increased.

Filtering by various means has been almost universally employed to "purify" water. Specifically, Harvey et al. (1944) and Messino et al. (1967) have found it effective in denucleating water. Centrifuging was reported (Harvey et al. 1944; 1945) to be effective in removing large macronuclei from water.

Degassing at room temperature under vacuum with sound or mechanical energy has been used (Harvey et al. 1944; 1945; Pease and Blinks 1947) with the idea that nuclei go into solution because of the very low gas tension of the water. However, Strasberg (1959) has shown that although the energy required for cavitation increases as dissolved air content decreases, the sound cavitation pressure at 0% air content is only about eight atmospheres, which is far lower than the theoretical estimates. Therefore, he concluded that nuclei containing dissolved air did in fact persist at very low dissolved air contents.

Boiling at 100 C has had wide application for degassing liquids, and some investigators claim denucleation (Harvey et al. 1944, 1945; Pease and Blinks 1947) by this method. It is important to note that perhaps the highest tensile strength values for water were obtained by Briggs (1950) using boiled distilled water containing an undetermined amount of dissolved air. Between 5 and 30 C, he developed tensile strengths of 240–275 atmospheres by centrifuging a Z tube of water.

In summary it can be said that all of the previously described methods appear to be at least partially successful in denucleating water in that after treatment, the water can be either heated to higher temperatures before boiling begins or the negative pressures required for cavitation at room temperature are increased.

EXPERIMENTAL

The experimental procedure was essentially the same as that described in the previous study (Gertjejansen and Hossfeld 1967). Pulp pads were formed from slurries of unrefined bleached southern pine kraft or unbleached western softwood sulfate. Prior to pad formation, the slurries were thoroughly deaerated by cavitating under vacuum to prevent bubble entrainment in the pad. Distilled water was used as the permeating fluid, and its age and dissolved air content were varied to suit the experimental needs. Dissolved air contents were measured with a Beckman Model 777 Oxygen Analyzer. Water was aged by storage in covered 5 gallon polyethylene or Pyrex jugs.

A head of 46.5 cm of water, measured to the nearest 0.1 cm, was used. To prevent pad compaction during flow, all pads were compressed to a density which was greater than that produced by the frictional pressure drop. Permeability coefficients were calculated from Darcy's Law.

$$Q = KA \cdot dP/uL$$

Where

Q = volume rate of flow, cm^3/sec
A = gross cross-sectional area of the porous medium, cm^2
u = viscosity of the fluid, dyne-sec/cm^2
L = length of the porous medium, cm
dP = frictional pressure drop across the porous medium, dynes/cm^2
K = permeability coefficient of the porous medium, cm^2

RESULTS AND DISCUSSION

(a) *Size classification of the causal agent in aged distilled water*

Estimates of the size of stable bubble nuclei in water have been made by acoustical techniques. In water containing minute bubbles, the absorption of sound energy is dependent primarily upon those bubbles which resonate at a frequency related to their size; the smaller the bubble, the higher the frequency required for resonance. Iyengar and Richardson (1958), using one-day-old tap water and employing frequencies up to 1,000 KHz, obtained peak attenuations at frequencies corresponding to bubble diameters of 7 and 12 micrometers. Strasberg (1959), using frequencies of 150, 250, and 550 KHz and tap water varying in age from fresh to 80 hr, found no significant attenuation after the water had stood for 3–4 hr. Therefore, he concluded that the nuclei must be smaller than 6 micrometers, the resonant radius for 550 KHz. Turner (1961) found that the attenuation of fresh tap water was significantly greater than that of distilled water at 5.125 MHz. He attributed this difference to nuclei of approximately 1.6 micrometer diameter, the resonant size for that frequency. However, this does not mean that nuclei were not present in distilled water because his measurement technique did not yield absolute attenuation values; they could have been more profuse in the tap water or of a different size in the distilled water. In addition, Turner did not elaborate on the age or condition of the distilled water; if the water was freshly distilled, a large difference in attenuation would be expected when compared to tap water.

Two other studies of nuclei size have been made using other techniques. Fox and Herzfeld (1954), by studying cavitation pressures at different frequencies, estimated that bubble nuclei 2–8 micrometers in diameter existed in water. Iyengar and Richardson (1958) used light scattering measurements and estimated that nuclei of 0.8 micrometer diameter existed in long-standing water. Thus all of the work on estimating nuclei size has been done on tap water, and a diameter range of 0.8–12 micrometers has been obtained.

In the present study, Millipore filters of different pore diameters were used to classify the size of the causal agent in aged distilled water. Two 142 mm filter holders were placed in parallel up-stream of the permeability cell, and filters of 0.22, 1.2, and 5.0 micrometer pore diameter were used. The pulp pads, which inherently contain wide ranges of pore sizes, served as

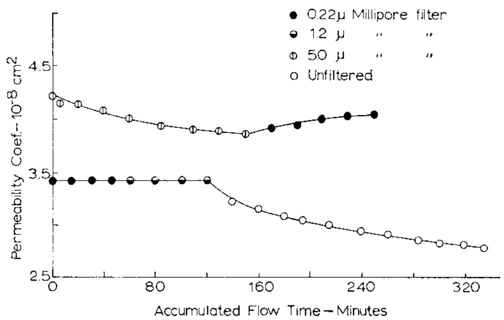

FIG. 4. Classification of the size of the blocking agent in aged distilled water using Millipore filters of different pore diameters.

indicators of the presence of blocking agents in the permeating water. A blocking agent small enough to pass through a filter would cause a decrease in pulp pad permeability. Fig. 4 shows the results obtained by permeating two different pulp pads of unbleached western softwood sulfate at densities of 0.154 and 0.159 g/cm^3. It is immediately evident that the 0.22 and 1.2 micrometer filters were effective in preventing a permeability decrease, whereas the 5.0 micrometer filter was not. Thus the size of the causal agent in aged distilled water was larger than 1.2 micrometers and smaller than 5.0. Since many bacteria fall within this size range, samples of aged water were agar plated, but the results were negative. Direct microscopic analysis of the filters showed that freshly distilled water, which does not cause a permeability decrease, contained approximately the same small number of particulate contaminants as did aged water, none being identified as bacteria.[2] Most important, the size range of 1.2–5.0 micrometers obtained with aged distilled water falls precisely within the size range obtained for bubble nuclei in tap water by other investigators (Fox and Herzfeld 1954; Iyengar and Richardson 1958; Strasberg 1959; Turner 1961). Bubble nuclei could be present initially in freshly distilled water and then grow and stabilize over time in association with the microscop-

[2] The microscopic analysis did indicate, however, that a still is not a purifier, and some foreign matter apparently can be carried over with the hot vapor.

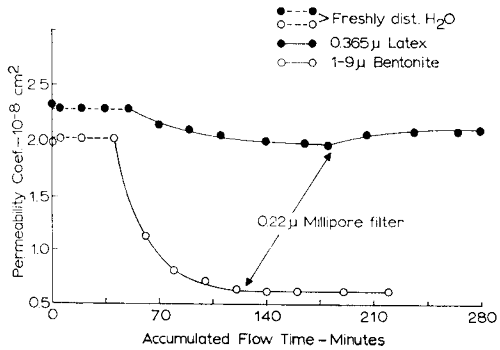

FIG. 5. The effect of two particle-water systems on the permeability of bleached southern pine kraft pulp pads.

ically visible contaminants, in association with submicroscopic contaminants, or independent of contaminants to produce the deleterious effect of aged distilled water on pulp pad permeability.

It is unfortunate that more acoustical work has not been done on distilled water, although Strasberg (1959) has made such a suggestion. It seems reasonable that a filtering technique, as employed in this study, would lend itself nicely to acoustical determinations of bubble nuclei size in all water types.

(b) Effect of solid particles on pulp pad permeability

In the previous study (Gertjejansen and Hossfeld 1967), it was shown that when a pulp pad had undergone a permeability decrease as a result of permeation by unfiltered aged distilled water, equilibrium was eventually attained, and subsequent insertion of a 0.22 micrometer filter resulted in a permeability increase to a new equilibrium somewhat less than the original pad permeability. This phenomenon is illustrated by the upper curve of Fig. 4. Since nuclei are theorized to be minute stable bubbles, an attempt was made to duplicate this behavior by permeating with water-particle suspensions. According to the results of Quincke (Abramson 1934) bubbles of air, oxygen and hydrogen, like cellulose, are electronegative relative to water; consequently two electronegative particle systems of different size and concentration were chosen: 0.365 micrometer diameter polystyrene latex particles at a concentration of 2 ppm by weight in freshly distilled water and bentonite clay particles, 1–9 micrometers in diameter at a concentration of 60 ppm. Fig. 5 shows the results obtained by permeating two pulp pads of bleached southern pine kraft with the two suspensions. The behavior of the small, low-concentration latex particles duplicates that of aged distilled water whereas the larger, more highly concentrated bentonite particles were capable of completely occluding almost all pad pores, and subsequent insertion of the 0.22 micrometer filter only resulted in a cessation of permeability decrease.

It had been previously postulated (Gertjejansen and Hossfeld 1967) that particulate contaminants in aged distilled water were not responsible for the permeability decrease since Megraw (1967) had shown that when wood permeability was decreased by permeation with a virus-water suspension, subsequent permeation with virus-free water did not regain the lost permeability. However, a pit membrane differs radically from a pulp pad, and when one considers the tortuosity of a pulp pad, particles probably do not always pass through or occlude pad pores but instead are capable of momentary occlusion in pores of somewhat larger average diameter. Eventual equilibrium indicates that, in addition to complete plugging of smaller pores and unobstructed passage through large pores, particles in pores of certain critical sizes leaving the pad are replaced by incoming particles at the same rate so that the net flow area remains unchanged. Subsequent insertion of the filter eliminates the incoming particles, and the continuing loss of outgoing particles increases the net flow area which in turn increases permeability. The net loss in permeability, that is the difference between permeation with freshly distilled water and equilibrium permeability obtained with the filter, is a measure of the flow area lost by complete plugging of the smaller pad pores by the latex particles. Since, as mentioned pre-

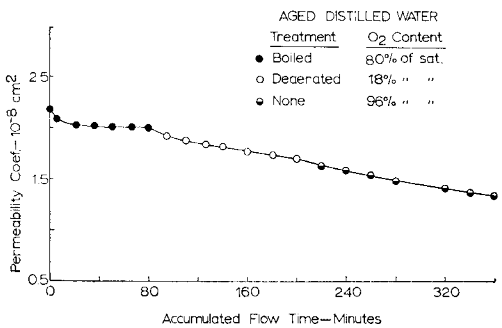

FIG. 6. The effect of boiled aged distilled water on the permeability of a bleached southern pine kraft pulp pad.

viously, the microscopic counts revealed that freshly distilled water contained approximately the same number of particulate contaminants as did aged distilled water, the latex suspension has then shown that the causal agent in aged distilled water behaves as a particle but does not appear to be one. Stable bubble nuclei would fit this description.

(c) Effect of boiling on aged distilled water

Since Briggs (1950) had obtained very high tensile strengths for boiled distilled water, it appeared likely that the boiling treatment could have altered or reduced bubble nuclei in the water, which in turn accounted for the high tensile values. By the same reasoning, a boiling treatment should then reduce the deleterious effect of aged distilled water on pulp pad permeability. Fig. 6 shows the results obtained by permeating a bleached southern pine kraft pad with boiled (100 C) aged distilled water followed by permeations with the same water not boiled but deaerated and then not deaerated. Deaeration was accomplished by induced cavitation from mechanical shock under vacuum at room temperature. The boiled water was cooled to room temperature prior to permeation.

It is evident that the boiling treatment definitely minimized the permeability decrease, whereas deaeration had very little effect. The suggestion could be made that globular proteins present in the aged water were denatured by the boiling treatment since it is well known (Putnam 1953) that many proteins will, upon heating, become less soluble, become aggregated, and then precipitate out of solution. Although this is possible, the origin of the protein would most likely be attributed to microorganisms, but none were revealed in the microscopic analysis or agar plating. An alternative explanation would be that the thermal energy was functional in reducing the size of bubble nuclei by the greatly increased diffusion coefficient at the high temperature or by breaking down the stabilizing mechanism.

The inability of deaerated aged distilled water to prevent a permeability decrease, as illustrated in Fig. 6, had been shown previously by Gertjejansen and Hossfeld (1967). Their results are in accordance with the findings of Strasberg (1959) which indicated that stable bubble nuclei were present at low dissolved gas content.

(d) Effect of water types on specific filtration resistance

In addition to the deleterious effect of unfiltered aged distilled water on pulp pad permeability, experimentation had shown that tap water produced similar but more drastic decreases. Thus a practical interest developed as to the effect of these two water types on the filtration resistance of pulp slurries. Using the permeability cell with an extension tube as a constant head filtration device (Gertjejansen 1964), the specific filtration resistance of an unbleached western softwood sulfate pulp was determined at three freeness levels using four water types: unfiltered aged distilled water, freshly distilled water, unfiltered tap water, and filtered tap water. The slurries were of 0.3% consistency and filtration was carried to a fiber deposition of approximately 1,000 g/m^2 which corresponded to a filtrate volume of 150 ml. Fig. 7 shows that there was no discernible difference in filtration resistance between the four water types at all freeness levels. Because of the permeability data, one would have expected the aged distilled water and unfiltered tap water to increase filtration

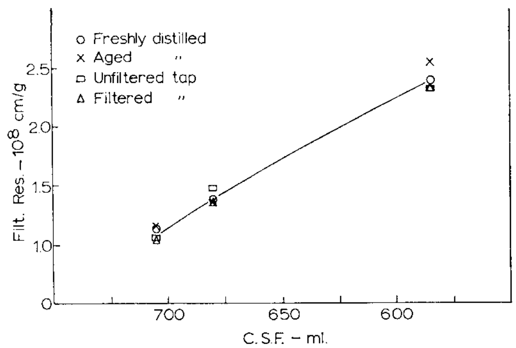

FIG. 7. The effect of four water types on the specific filtration resistance of an unbleached western softwood kraft pulp at three Canadian Standard Freeness (C. S. F.) levels.

resistance; the fact that they did not is probably attributable to two factors. First, at the flow rates used in the permeability experiments, only five minutes would have been required to pass the quantity of white water (150 ml) used in the filtration resistance determinations. This time interval would correspond to an approximate permeability decrease of only 0.05×10^{-8} cm^2. Thus insufficient volume of filtrate was involved to cause a discernible filtration resistance increase. Second, the pad formed by filtration at 60 cm of water, the head used in this experiment, has a lower density and consequently larger pores than those pads employed in the permeability experiments. The pad density at that compacting pressure would be only 0.10–0.12 g/cm^3.

SUMMARY AND CONCLUSIONS

The present study, in an attempt to define better the properties of aged distilled water as related to pulp pad permeability, has shown the following results.

1) The size range of the blocking agent in aged distilled water responsible for pulp pad permeability decreases falls precisely within the size range obtained for bubble nuclei in tap water.

2) Latex particle suspensions in freshly distilled water displayed a behavior identical to that of aged distilled water, yet there was no detectable difference in the counts of particulate contaminants in freshly distilled and aged distilled water.

3) Permeability decrease was greatly reduced by boiling aged distilled water prior to permeation, whereas deaeration at room temperature had no effect.

Thus we see that the water treatments and subsequent pad behavior definitely point to a blocking agent that behaves as particulate matter yet does not fit that description in every respect. Bubble nuclei would explain the permeability results obtained in this study, but how they are stabilized and why aging promotes their growth and existence are still matters of conjecture. Although pulp pad permeability was affected by aged distilled water, it was shown that the specific filtration resistance of pulp slurries, a consideration of practical importance, was not increased by either aged distilled water or tap water when compared to freshly distilled water and filtered tap water.

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