

ANOMALOUS DECLINES IN LIQUID PERMEABILITY

N. Bona, M. Anelli and M. Rosania
Eni E&P

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ABSTRACT

We discuss single-phase flow experiments, in which fully saturated core samples are flowed for a long time (months) with the saturant liquid at extremely low rates. Absolute liquid permeability is seen to decline during flowing, but Klinkenberg permeability recovers after cleaning and drying the sample.

We see that:

- Water permeability declines more than oil permeability
- Undegassed liquids are more damaging than degassed liquids
- The application of an elevated backpressure reduces the permeability loss.

We show that:

- Fines movement and other classic damaging mechanisms are not entirely compatible with our observations
- The analysed samples must have suffered from a sort of embolism caused by microbubbles of gas in suspension in the liquid
- Naturally occurring cavitation nuclei can explain all the results.

EXPERIMENTAL

The tests presented in this paper were performed on two sandstone samples. One sample (#56) is medium grained, with a Klinkenberg permeability above 400 *md* and a porosity of 15%; the other sample (#102) is fine grained, with a Klinkenberg permeability below 1 *md* and a porosity of 7.5%. Both samples have a poor to moderate degree of sorting and a grain-cement supported texture. The detrital assemblage is made up almost exclusively of quartz grains. The most significant authigenesis is silica as quartz overgrowths and represents the major control of permeability. The following procedure was used.

1. The samples were cleaned and dried in a humidity oven.
2. Klinkenberg permeability was measured under a hydrostatic confining pressure of 870 *psi*, by interpolating a 3-point nitrogen permeability curve.
3. The samples were saturated with synthetic formation brine filtered to 2.7 μm (NaCl salinity of 100 *g/l*) by evacuating for 8 *hr* and pressurising to 2000 *psi* for 4 *hr*.
4. The samples were mounted in a vertical core holder, hydrostatically pressurised to 870 *psi*, and flowed for one month with the saturant brine at extremely low rates (Darcy velocity did not exceed 15 *cm/day*).
5. The pressure difference across each sample was monitored on a daily basis.
6. The samples were dismantled, cleaned and dried in a humidity oven.

7. Klinkenberg permeability was re-measured.
8. The entire cycle was then repeated using another liquid or the same liquid but with a backpressure imposed on the sample. Typically, each sample was tested with undegassed water, degassed water and degassed Soltrol oil, at two backpressures (ambient and 2000 *psi*).

RESULTS

Fig.1 and **2** illustrate the results. **Fig.1** shows the entire sequence of experiments in terms of measured permeability versus elapsed time. The curves show that the liquid permeability declines; the Klinkenberg permeabilities measured before and after each flow test correspond to the isolated points. Each test lasted approximately one month, and two different injection rates were used in most of them, as shown in the lower part of the plot. **Fig.2** illustrates the observed declines in liquid permeability expressed in terms of percentage of initial permeability. The following observations are noted:

1. Liquid permeability declines during flowing.
2. The permeability for undegassed water declines more than that for degassed water, and the permeability for degassed water declines more than that for degassed oil.
3. The greatest decline in permeability takes place in the highest permeability sample.
4. During flowing at ambient pressure (*i.e.* with atmospheric backpressure), the permeabilities for all liquids decline much more than with 2000 *psi* backpressure.
5. At ambient pressure, permeability never reaches stability, while stability is achieved at 2000 *psi*.
6. Klinkenberg permeability is recovered after sample re-cleaning and drying.

DISCUSSION

Fines Movement. Although the literature does not report a large number of long-term flow experiments, there are other cases where absolute permeability to water is seen to decline during flowing but recovers with drying and resaturation. In tight sands, for example, this behaviour is common and is attributed to a combination of fines migration and delayed hydration. In practice, it is assumed that delicate clay minerals are mobilised during flowing, but re-attach in more favourable locations after sample drying, when they no longer have a hydration water layer¹. Water permeability declines have also been observed in rocks similar to our samples, namely in a Berea with 19.4% porosity and 259 *md* air permeability consisting of quartz overgrowth cemented sandstone with minimal grain-coating clay². In this experiment, deionized water was flowed at a high Darcy velocity (around 17 *m/day*), and water permeability was seen to diminish by a factor of 3.7. SEM analyses attributed this permeability decline to kaolinite movement and brushpeeling (reversible movement of clay filaments). Another study reporting water permeability declines in rocks with little clay content was performed by Gash³ on coal samples submitted to two-month flowing tests with deionized water. This work is interesting because very low Darcy velocities were used (on the order of 4 *cm/day*) and nevertheless water permeability decreased by an order of magnitude. Again, fines movement was regarded as the main cause of the permeability loss. In Gash's paper, the restoration of liquid permeability following flow reversal was taken as the principal evidence of fines migration. However, it is clear that other kinds

of impurities in suspension in the water (such as microbubbles of gas) could produce similar effects on permeability.

Although fines movement, be it mechanically or chemically induced, is often called on to explain a permeability decline, it cannot explain the results reported in the present paper. We see that putting a backpressure on the sample does make a difference, and this pressure dependence cannot be ascribed to solid particles (whose sizes are independent of pressure). Mechanically induced fines migration is reported to occur for interstitial velocities greater than a critical value ranging from 0.03 *cm/s* to 0.44 *cm/s* depending on the authors^{4,5,6}, and we used velocities that are more than a factor of 10 lower than the lowest of these values. Low water salinity can cause swelling clays to exchange ions, expand and possibly detach from host grains, but for water salinities greater than 20 *g/l* NaCl these mechanisms are unlikely to occur. In our experiments, clay-water interactions must be excluded because they are not consistent with the very small amount of clays in the analysed samples and the relatively high salinity (100 *g/l* NaCl) of the saturant brine. Moreover, they do not explain why degassed and undegassed waters with identical chemical compositions have different effects on permeability.

Other Damaging Mechanisms. Besides fines migration, which is thought to be the major source of permeability damage in published studies, other concurring mechanisms include scales deposition, precipitation of newly formed materials and bacteria formation. These processes cannot explain our results either. Bacteria may act as impurities in suspension in the water and have sizes of the order of tens of microns, so in theory they can affect permeability. However, the concentrations of bacteria in tap water (all our waters were prepared from tap water) are very low if not zero, and in some experiments we added bactericides such as sodium hypochlorite (NaOCl) or sodium azide (NaN₃) to the water composition. In order to investigate the occurrence of salt or cement precipitation, we took “twin” samples close to the analysed samples and flowed them under identical experimental conditions. Then we made SEM observations of small chips taken from the inlet and outlet faces of these samples both before and after flowing. Although minor scales were seen in one of the samples, their intensity and spatial extension cannot explain the observed permeability declines.

Gas Bubbles. We believe that slowly developing gas saturation is the primary cause of the permeability loss seen in our experiments. Gas bubbles suspended in the flow are the only thing that can reduce rock permeability reversibly (damage disappears after drying), and in a way that is dependent on the pressure of the liquid and the degree to which the liquid has been degassed. Gas bubbles may be either artificial or natural. Natural bubbles are discussed later. Artificial bubbles originate from experimental problems such as the injection of air during flowing owing to a micro-leakage in the pump circuit or the presence of air trapped in the flow-line. Other kinds of unwanted gas in the sample include pre-existing air or CO₂ as a result of non-optimal saturation procedures, and air that devolves from liquids that have not been degassed adequately. Air bubbles can explain why permeability to water declines more than that to oil. This observation is consistent with the fact that air is more soluble in oil than in water: according to Battino *et al.*⁷, at 25°C and atmospheric pressure, one litre of a C₁₀ - C₁₃ mixture (these hydrocarbons should reflect the characteristics of Soltrol, the oil used in our experiments) can dissolve 5.3·10⁻³ moles of nitrogen, while one litre of 100 *g/l* NaCl brine can dissolve only 3.6·10⁻⁴. Degassed liquids can dissolve higher amounts of gas,

and that explains why permeability to degassed water declines less than permeability to undegassed water. Air bubbles explain also the observed dependence of permeability on pressure: as pressure increases, the solubility of air increases and the sizes of undissolved bubbles decrease. Finally, the presence of air bubbles in the liquid explains why the greatest decline in permeability is seen in the higher permeability sample: the two samples discussed in this paper were flowed at similar velocities. Because of their permeability difference (sample #56 has a Klinkenberg permeability above 400 *md*, while that of sample #102 is below 1 *md*), during flowing the average liquid pressure in sample #102 was 10^2 to 10^3 times greater than that in sample #56.

Creep Effects. The results show that permeability declines rapidly during the first hours. That is probably the effect of creep, that is the compaction of the sample due to the applied confining pressure. Only after this period does permeability start to be controlled by the dynamics of gas bubbles. It is interesting to note that Gash³ notices the same phenomenon. In his experiments, confining pressures vary between 450 and 1000 *psi*, and during the first 24 hours water permeability is seen to lose approximately 50% of its initial value. After this period, the permeability decline does not appear to be associated with changes in the pore volume of the sample, because the porosity does not change even though the permeability continues to decrease significantly.

On Current Laboratory Procedures. The experiments described in this paper are part of a larger program in which various types of rocks were studied. We made tests also on glass filters. In all cases, we obtained results analogous to those that are presented here. The analyses have been performed using standard procedures and up-to-date equipment, and a commercial laboratory replicated part of the experiments finding the same things. In the light of that, one conclusion that might be drawn is that the procedures and equipment currently used by laboratories do not guarantee a truly full saturation of samples or the injection of truly air-free liquids. As long as injection tests take a few days, air bubbles should not represent a problem, but in longer tests, they can play an important role. The simplest way to avoid air bubbles is to increase the pressure in the sample by putting a backpressure on it. This expedience should be used also in ambient conditions tests. Because many published data from long-term liquid injections are obtained on samples open to atmosphere, we believe there could be a risk that the permeability declines reported in these studies are the result of accidental bubble injection and not fines movement as it is generally reported by their authors.

THE “CAVITATION NUCLEI” HYPOTHESIS

Artificial air bubbles inadvertently injected into core samples can explain the results we obtained. But there is another type of bubble that might explain our results. These bubbles are the so-called cavitation nuclei. This section introduces cavitation nuclei and reviews the theoretical and experimental work published on this subject.

When a gas is dissolved in a liquid, a small fraction of the gas is held as free microbubbles. The presence of these microscopic gas bubbles, or nuclei, somehow stabilized in liquids, has been hypothesized by physicists to explain the fact that the observed nucleation threshold in certain liquid-gas solutions is much less than that predicted by classical thermodynamics. “Nucleus” in this sense refers to agglomerations of gas or vapour molecules of sufficient size (microns) to allow later growth, upon the imposition of additional heat energy or reduced liquid pressure, into conventional

bubbles. In the absence of nuclei, boiling, cavitation and solution gas liberation under the relatively low superheat or under-pressure conditions under which they are commonly observed, could not exist. As an example, ultra-pure water can sustain temperatures above 300°C at atmospheric pressure before a vapour bubble can be generated in it^{8,9}.

Nuclei Formation. Probably the most important mechanism for the generation of nuclei is that of gas entrainment, which may occur wherever a free water surface is present. The high level of entrained micro-bubbles in normal tap water presumably results from the effect of such free surfaces as found in storage reservoirs. There is an even higher level of gas entrainment in the sea and other large bodies of water. Aquifer waters do not make an exception, as they are or have been in contact with hydrocarbon gas or air for a long time in their life. Extremely small microbubbles can also be created by thermal fluctuations. Another mechanism for the generation of nuclei is the interaction of water with cosmic radiation. This was first suggested by Sette *et al.*¹⁰, who compared nucleation pressure threshold variation in a beaker with and without external lead shielding. Cosmic rays (in particular μ -mesons) have penetration depths on the order of thousands of metres, so their action should be felt not only in the laboratory but also by aquifer waters.

Nuclei Stabilization. Whatever the generation mechanism may be, there must be a stabilization mechanism that prevents the formed nuclei from rapid collapse and disappearance. The simplest model for a nucleus is that of a microscopic gas bubble present in the bulk of the liquid. But a free gas bubble is inherently unstable, that is, it will shrink and dissolve very quickly¹¹. According to Epstein¹², free bubbles of size on the order of a few microns, dissolve in a few seconds even if the liquid is supersaturated with gas. Larger bubbles live longer, but they form only under particular local environmental conditions which cause the microscopic nucleus to grow instantaneously. This is not the case of ordinary laboratory flow tests where water is normally undersaturated (typically, water is in equilibrium with air at atmospheric pressure, and during flowing the pressure in the sample cannot be lower than this value). Thus, nuclei must be stabilized in some way. Two models are found in the literature.

The first model considers gas nuclei trapped in the asperities of pore walls or in microcrevices in the surfaces of solid particles suspended in the liquid^{13,14,15}. The stabilization of the gas is assured by a meniscus concave toward the liquid (the opposite curvature would lead to complete extinction of the gas bubble). The second model considers gas nuclei in suspension in the liquid. The stabilization of the nuclei is achieved through a film that wraps the nucleus. If the liquid pressure increases, the nucleus shrinks, but the film is supposed to turn into a skin that prevents diffusion of gas and stops the nucleus from shrinking further, that is, it is stabilized. Fox¹⁶ hypothesized that the film is composed of organic impurities such as fatty acids. An alternative model¹⁷ assumes that nuclei cannot shrink below some lower limit because they carry identical surface charges (ions) that generate a repulsive force. Later experiments¹⁸, however, led to exclusion of this model. More recently, Yount¹⁹ interpreted the stabilization of nuclei as the result of a skin of variable permeability composed of surface-active substances of amphiphilic nature. From experiments, Yount²⁰ estimated that the skin thickness is on the order of a few nanometres. On exposure to reduced liquid pressure, the nucleus expands and acquires a 2-component surface, made up of surfactant molecules identical in area with the stabilized nucleus surface, and of

liquid molecules (**Fig.3**). According to this model, the effective interfacial tension of the nucleus, γ_{eff} , is a variable quantity which approaches that of a free gas bubble without a skin as the radius of the nucleus increases. Stabilized nuclei, on the other hand, have vanishing interfacial tensions.

Experimental Observations. Both trapped and suspended nuclei may exist in water bearing rocks, but only the latter can explain our laboratory results. Therefore, we will deal with this second form of nuclei. Several techniques to measure the distribution of stabilized nuclei suspended in water are reported in the literature. These include the Coulter counter, holography, light scattering methods, cavitation susceptibility meters and acoustic methods. **Fig. 4** is a compilation of experimental data for natural water²¹. The figure shows the number density distribution, $n(R)$, in m^{-4} , as a function of the nucleus size, R . Quantity $n(R) \cdot dR$ represents the concentration (number per unit volume) of nuclei with sizes from R to $R+dR$. As can be seen, over the range of sizes in which the measurements have been made (1 - 100 μm), the distribution function appears to be a power law of the form $n(R) = B/R^4$. The magnitude (B factor) of the function appears quite different from one set of data to another: B ranges from 10^{-10} (tap waters which have been standing in a tank for considerable time) to 10^{-7} (sea waters). The number of nuclei in the 1-100 μm size range in a unit volume of water correspondingly varies between 30 *nuclei/cm³* for tap water and $3 \cdot 10^4$ *nuclei/cm³* for sea water. Direct measurements of nuclei density were made also by Liu and Brennen²² in the Low Turbulence Water Tunnel at Caltech. They used water filtered to 5 μm and observed values in the range of 100 - 250 *nuclei/cm³*. Slightly different results were obtained by Johnson and Cooke²³, who observed nuclei in filtered seawater at atmospheric pressure within a range of sizes from 0.7 to 13.5 μm . Johnson and Cooke's experiments are important because they proved that nuclei are stabilized by a skin: at exposure to large pressures, they saw that bubbles tended to dissolve leaving transparent remnants. Stabilized gas nuclei were later observed also by Yount *et al.*²⁰ in distilled water, with sizes on the order of 1 μm .

NUCLEI AND EXPERIMENTAL RESULTS.

Damaging Potential. In this section, we reconsider our experiments in terms of cavitation nuclei. The first thing we want to note is that nuclei sizes are of the same order of magnitude as pore thresholds. The amount of nuclei in tap water is not high. According to **Fig.4** their density does not exceed 100 *nuclei/cm³*. This number might appear insufficient to explain the observed permeability declines, but it should be noted that in a laboratory flow test the analysed sample is continuously supplied with new nuclei from the injected water. Part of the injected nuclei will remain in the sample as it acts like a filter. Thus, the concentration of nuclei in a long-term flow test is expected to increase with time. Consider, for example, a laboratory water that contains 30 *nuclei/cm³*. If this water is injected into a 1.5" diameter sample at a Darcy velocity of 10 *cm/day* for one month, then more than 10^5 *nuclei* will hit the inlet face of the sample during this period. These are more or less the conditions under which our experiments were conducted, and the number of involved nuclei looks quite large to us.

The damaging potential of nuclei is inversely proportional to their ability to deform. As is shown in **Fig. 5**, a nucleus will pass through a constriction only if it can change shape. In doing so, the surface of the nucleus grows, and its effective interfacial tension γ_{eff} correspondingly rises. Mørch²⁴ gives the expression $\gamma_{\text{eff}} = \gamma[1 - (A_0/A)]$, where γ is the

free gas-liquid interfacial tension, A_0 is the initial area of the nucleus, and A is the area of the expanded nucleus. For a constriction consisting of a smooth cylindrical conduit with, say, half the nucleus radius, A reaches values from $1.15A_0$ to $1.5A_0$ depending on the length of the conduit, and the effective interfacial tension γ_{eff} will consequently range between 10 and 25 *dynes/cm* (we assume $\gamma = 72$ *dynes/cm*). These relatively high γ_{eff} values imply that nuclei do not behave too differently from free gas bubbles, and would support the idea that they can really act as pore blocking agents despite their initial zero interfacial tension. A complication, however, arises from the fact that the skin of an expanded nucleus breaks up into islands of surfactant molecules separated by water (**Fig.3b**) and interfacial tension is zero on these islands¹⁹. Because of this, in theory the islands can support small local radii of curvature and might behave as the joints of an articulated system. Therefore, it would be possible that the nucleus conforms to the constriction while keeping its free gas-water interfaces essentially flat. In this case the capillary pressure would remain close to zero, the associated capillary number would consequently be high, and as a result the nucleus would not be able to clog the flow. This is a possibility that deserves deeper investigation, as will the breaking of nuclei into smaller bubbles and the extent to which this can happen. Anyway, various authors have seen that nuclei can be removed by filtering the water (Mørch²⁴ and the references therein). This fact represents an experimental proof of the ability of nuclei to occlude small pores (Note that at exposure to atmosphere nuclei reform in a relatively short time. A substantial lack of nuclei for bubble nucleation is to be expected only in very special cases where extraordinary degrees of gas removal and liquid purity are maintained. These cases represent the exception rather than the rule).

Effect of Gas Content. Published measurements of the concentration of nuclei in water show large variations depending on the quality of the water. Indeed, water source and history are very important factors. Bajic²⁵ shows that the magnitude B of the nuclei size distribution function increases with the total gas content of the water. This means that there are more nuclei in waters with more gas dissolved in them. This fact is consistent with our observation that permeability declines more with undegassed water than with degassed water.

Effect of Pressure. As reported, parts of our experiments were performed by putting a backpressure of 2000 *psi* on the outlet face of the sample. In these tests nothing but pressure changed. So the pressure differential (Δp) across the sample and the velocity of the flowed liquid remained the same. In the back-pressurised cases, permeability declined significantly less than in the corresponding experiments carried out without backpressure. In order to explain this result in terms of nuclei, a molecular argument developed by Franklin²⁶ is helpful. This author distinguishes between two kinds of dissolved gas molecules: molecules that have some freedom to move in the interstices of the liquid structure, come together to form clusters and constitute nuclei; and molecules that are locked into the local liquid structure. Only the latter can be considered as truly “in solution”. Any effect which reduces the extent of the local liquid structure, for example the increased molecular vibrations arising from a rise in temperature or a pressure reduction, would cause the proportion of mobile gas molecules to increase, and the concentration of nuclei would consequently rise. As a result, the nuclei size distribution will shift towards larger sizes. Experimental results supporting this dependence of nuclei size distribution on pressure are reported by Johnson and Cooke²³. In one of their experiments, the application of a pressure of only

0.081 *bars* reduces a population of stable nuclei in seawater by 74%. Nuclei 0.75 to 2.25 μm in size are reduced in number by a factor of 3, while those greater than 5 μm are reduced by a factor of more than 10. A further increase in pressure to 0.135 *bars* results in the collapse of all remaining stabilized nuclei below 0.5 μm .

Water vs Oil. In our experiments water permeability declines more than oil permeability. This result is consistent with the fact that there are a limited number of cavitation nuclei in oil. A recent study supports this argument²⁷. The authors state that serious doubt was raised about the “theory that minute bubbles called cavitation nuclei are omnipresent in liquid when it was experimentally discovered that a tensile wave in an ordinary oil hydraulic pipe is propagated at the same velocity as a compressive wave”. Franklin²⁶ shows that the magnitude *B* of the nuclei size distribution function is strongly dependent on the number of gas molecules in the biggest cluster incorporated into the liquid structure. A slight increase in this number might translate into a considerable reduction in *B*. If the extent (length-scale) of the local molecular structure of oil is greater than that of water (*i.e.* if oil is more structured than water), then a lower concentration of nuclei in oil has to be expected. That would explain why permeability declines less when the saturant liquid is oil.

CONCLUSIONS

The classical sources of permeability damage do not explain the anomalous liquid permeability declines observed in the laboratory flow tests described in this paper. The way liquid permeability declines is dependent on the liquid type, its local pressure and the amount of dissolved gas. A commercial laboratory replicated the experiments and obtained analogous results. All the observations would indicate that microscopic bubbles of gas suspended in the liquid play a primary role. It is possible that these bubbles have an artificial origin, and this would imply a need for better laboratory standards (equipment and procedures) in long-term flow experiments. However, also cavitation nuclei can explain the obtained results. Published data about the concentration of nuclei and their size distributions in water and mineral oil, as well as the effect of liquid pressure on these quantities, are consistent with our observations.

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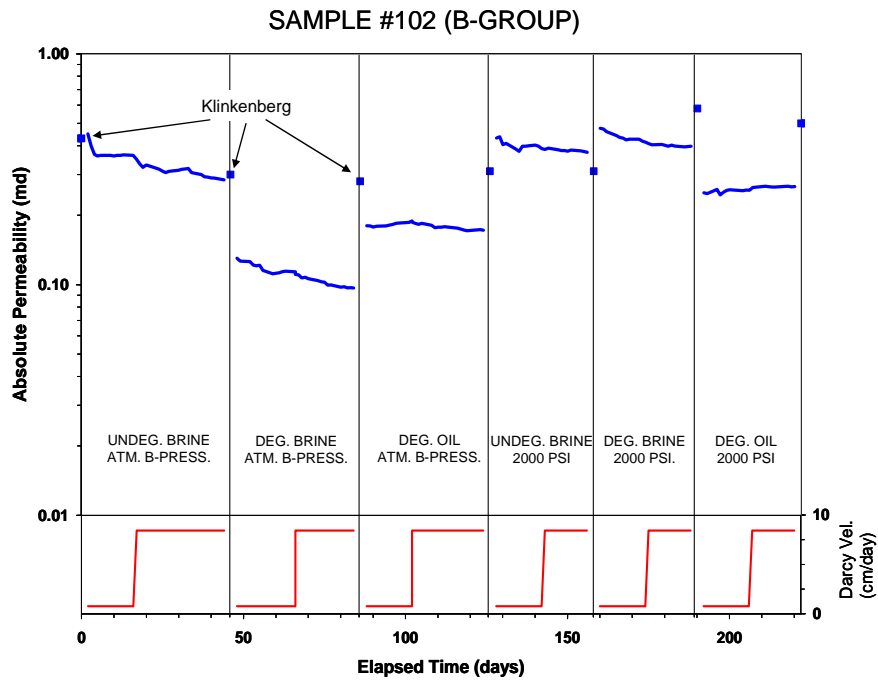
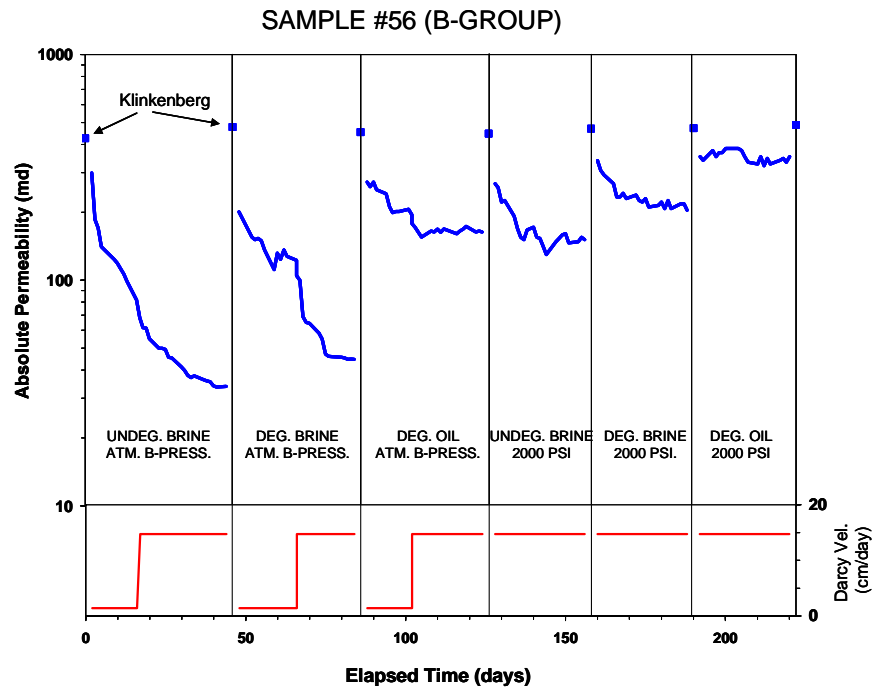


Fig.1 – Klinkenberg permeabilities (dots) measured before and after each flow test, and liquid permeability declines registered during flowing with different liquids and backpressures. Darcy velocities are shown below.

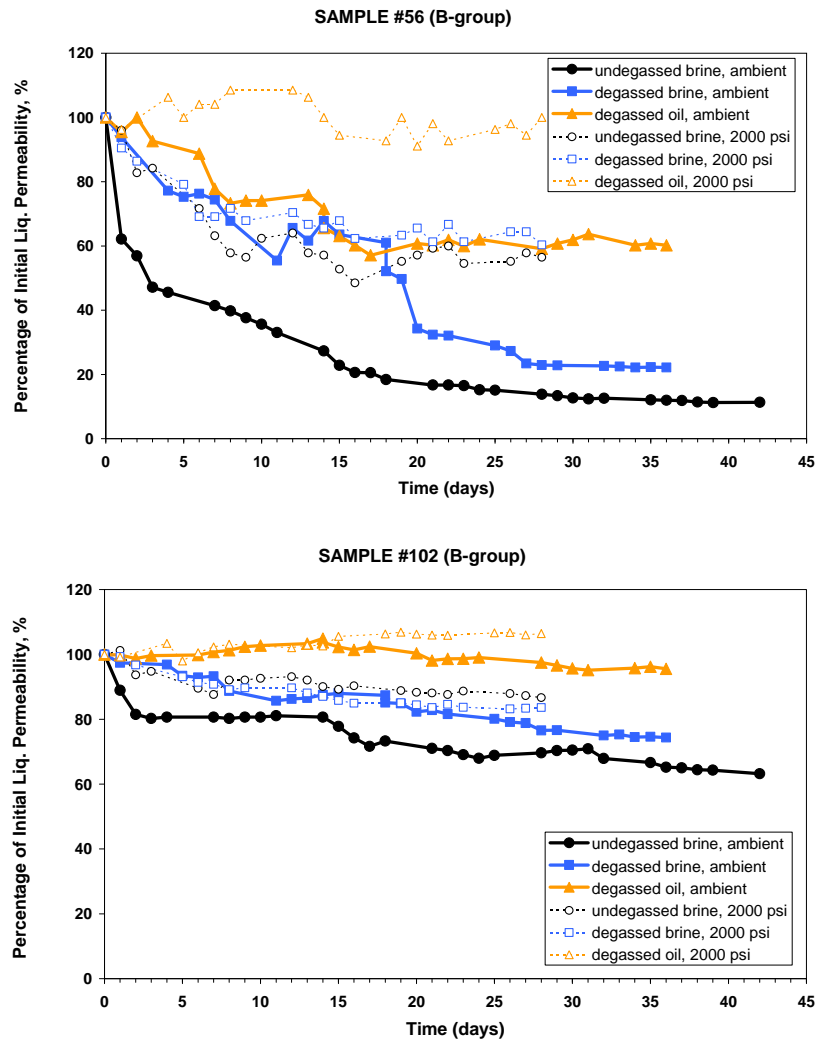


Fig.2 – Observed liquid permeability declines, expressed as a percentage of initial liquid permeability.

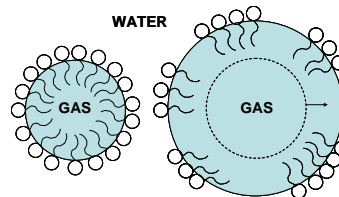


Fig. 3 Left: stabilized gas nucleus in suspension in water, fully covered by a monolayer of skin molecules causing the interfacial tension (IFT) to be zero. The surface area is A_0 . Right: expanded nucleus at exposure to reduced water pressure. The surface area is A . The skin breaks up into islands separated by free gas-water interfaces. IFT remains zero in the islands, but equals the water surface tension γ in the free gas-water interfaces. The average IFT of the expanded nucleus is $\gamma [1 - (A_0/A)]$.

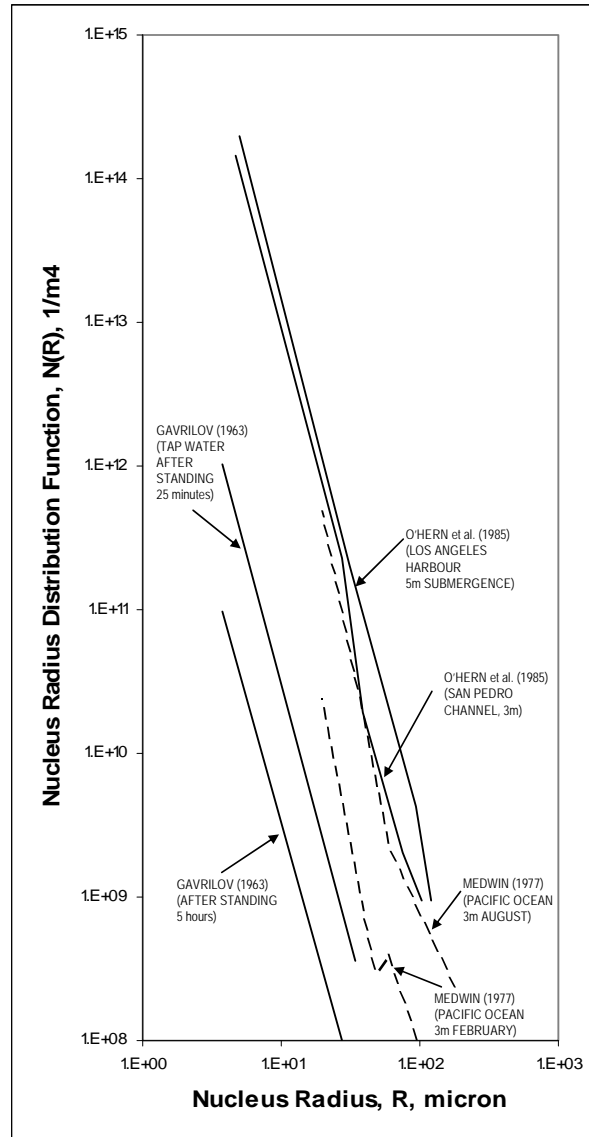


Fig.4 – Measured nucleus radius distribution functions for natural waters (adapted from Franklin, 1992)

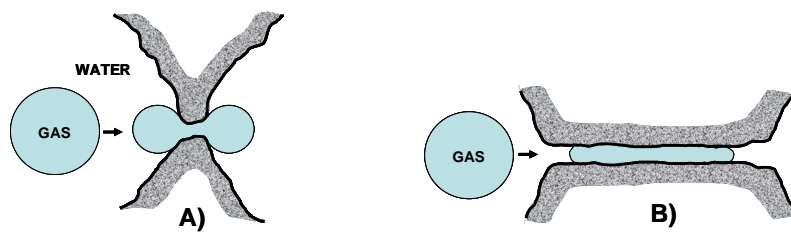


Fig.5 – Nucleus passing through a short (A) and long (B) conduit.