

IMPACT OF JOINT-SEALANT FAILURE ON THE COHERENCE OF PIPE-SEGMENTS COMPOSING WATER TRANSMISSION LINES: A CASE STUDY¹

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ABSTRACT

A commercial product used as pipe-joint sealant along the Shagra lateral pipeline, effected a repeating pattern of failure in form of cracks, water leakages, cement-lining fallout and severe corrosion. This study aimed to assess the stability and coherence of the sealant texture, and evaluate the possibility of release of potentially toxic contaminants from sealant into transmitted aqueous stream. The assessment was carried out as a laboratory bench test under simulated field conditions bracketing seasonal high and low temperatures. A portion of the powdered product was wetted and homogenized into a coherent paste, allowed 24 hrs to dry-set (harden), then soaked in distilled water for variable time lapses. At both temperatures, and for different exposure times, the set-sealant material suffered from distinct deterioration when brought into contact with water. Despite this, analysis of aqueous medium for toxic elements, (viz. Arsenic, cadmium, copper, chromium, lead, mercury, nickel, selenium and zinc), using EDAX, ICPEs, AAS-flame, -VGA & -GT, revealed complete absence of any significant leech of any of these elements. Conversely, GC amenable organic contaminants, probed with GC/MS, were completely non-detectable. In contrast, appreciably high chloride ion concentration, probed argentometrically, was found in the aqueous leechates. Dissolved TOC leaching from hardened sealant material, at ambient temperature, showed fairly low concentration (~2.5 ppm) compared to a corresponding, fairly high, TOC value (~58 ppm) at elevated temperature. The major constituent of this dissolved TOC was found to be starch. Finely powdered graphite particulates appeared to adsorb heavily on starch, which, in low temperature aqueous leeching solutions, seems to be released in form of micro-granules. Adsorbed layers of graphite particulates appeared to leach out together with these starch granules, which

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were observed as black residues trapped on membrane filters. For these reasons, it has been inferred that the presence of starch and graphite together, probably represents a major setback in the composition of the sealant material. The finite solubility of starch, and its distinct adsorbing affinity towards powdered graphite, seems to operate synergistically to weaken - together with chloride - the coherence of the set-sealant material. This behavior appears to have been the major cause of the repeated cracking of pipeline segments, the fallout of cement-lining and the severe corrosion of the underlying steel near pipe-joint zones of the Shagra pipeline.

1. INTRODUCTION

This project was initiated to test presence and evaluate concentration levels of potentially toxic chemicals that might possibly contaminate pipeline potable water by leaching out of pipe sealant product known as “X-Pando special No. 2”. The sealant is produced by X-Pando Products Company, Trenton, NJ, USA. The issue of the sealant was raised because in the Shagra pipeline branch of Al-Ghasim/Al-Rheyadh Project, a repeating pattern of severe corrosion, line breaks and leaks were noted localized mostly on pipe-joint zones, all along the line, where the above mentioned product was used as a joint sealant [1]. The company submitted representative samples for chemical analysis, together with a detailed literature of the product including documentations of its basic constituents [2]. The present study was carried out in verification to that end.

2. OBJECTIVES

1. To determine and evaluate the concentration of potentially toxic inorganic and organic constituents in the pipe-joint sealant “X-Pando special No.2”. The inorganic constituents to be monitored, will include the following toxic metals: mercury, arsenic selenium, cadmium, lead, nickel, chromium, copper and zinc; in addition to chloride, sodium and magnesium.
2. To investigate the possibility of sealant-leached organic components into the aqueous medium, probed e.g. by TOC measurements, or otherwise; at both ambient and higher temperatures and in presence and absence of chlorine residual (in the order of ≥ 0.5 to ≤ 1 ppm).
3. To monitor the integrity and coherence of the sealant as it comes into prolonged contact with water at ambient and elevated temperatures, and how could the

presence of chlorine residual affect the integrity of the product, or possibly lead to formation of undesirable chlorinated by-products with sealant-released organics.

4. To explore the possibility of release of solid particulates from the set product into pipeline potable water, probed as suspended solid load in the aqueous leachate of hardened sealant as a function of time; at both temperatures, and presence/absence of chlorine residual
5. To carry out, if necessary, further characterization of any other indicated potential contaminant, as appropriate. This will be based on identity, magnitude, and concentration levels of detected organic and inorganic entities.

3. METHODOLOGY

3.1 *Sample Treatments and Analysis*

3.1.1 *Original Product Sample*

Duplicates of intact, fresh factory sample, in the powder form, was digested in HCl, HNO₃, HClO₄ and HF in Teflon bombs, evaporated to near dryness and made to volume with 2% HNO₃ acid. Analysis of the digestate for toxic elements was carried out using ICPEs and/or AAS with oxy-acetylene flame, N₂O-acetylene flame, VGA or GTA.

3.1.2 *Soluble Product Aqueous Sample*

About 100 g of accurately weighed portion of powdered X-Pando product was placed in a liter of distilled deionized (DDI) water, covered with Para-film and allowed to stand overnight. After 24 hrs the supernatant aqueous layer was carefully decanted and vacuum filtered on a 0.2 μm cellulose nitrate membrane filter. The filtrate was then analyzed for dissolved metals using the same techniques mentioned above.

3.1.3 *Preparation of Hard-Set X-Pando Lumps*

A paste of the product was prepared by wetting about 500g of the powdered sample with successive aliquots of DDI, totaling a final volume of a 100 mL. These quantities, in proportion of 5:1, were in accordance with the company recommendation for making a relatively firm, “peanut butter” like paste [2]. The paste was promptly

poured in a plastic-designed forum of an ice-cube maker. The wet X-Pando fillings were allowed to stay overnight (24 h) in order to harden and set [2]. Fourteen hard-set, fairly uniform in dimensions, cube-like replicates were obtained, with an overall surface area amounting to $\sim 20 - 22 \text{ cm}^2$ per cube.

3.1.4 Preparation and Analysis of Ambient Temperature Leachates -1, -2 & -3 for Released Metals and Particulate Suspended Solid Analysis

Duplicates of the set product were soaked in a one liter beaker of DDI water for repeated progressive leaching time lapses. Leachate-1, was obtained after soaking the replicates for 24 hours, collecting the aqueous leachate-1 by decantation, and re-soaking the same replicate cubes in fresh one-liter volumes for another 24 hours period. Again, by decantation after the 2nd 24 hrs period a 2nd leachate (leachate-2) was obtained. Leachate-3 was similarly obtained by replacing the decanted leachate-2 by a 3d fresh one-liter volume for each cube and decanting, but this time after 48 hrs. Each of the three leechates was filtered at the end of each period, as above. In each case the DDI extractant (leaching DDI) was stirred at a very slow rate, barely enough to quietly swirl the aqueous phase and prevent stagnation. Each of the filtrates was then analyzed, as explained above, for toxic elements; whereas the residues in the pre-weighed membrane filters were dried to constant weight for gravimetric determination of the detritus load of suspended particulate matter.

3.1.5 Preparation of Higher-than-Ambient Temperature Leachates for Analysis of Metals Land Particulate Matter Release at Different Leaching Times

Duplicates of hard-set X-Pando cubes were allowed to leach for 24 hrs. at ca. $45 \text{ }^\circ\text{C}$ ($45 \pm 3^\circ\text{C}$). The resulting leachate was analyzed, as before, toxic metals and suspended solids. A similar leaching experiment was carried out at the same temperature ($45 \pm 3^\circ\text{C}$), but for only 6 hrs, in order to assess shorter term, high temperature, effect on leaching of metals & suspended particulate matter, as well as the physical stability and coherence of the product after setting.

3.16 Chloride Ion Analysis

Chloride ion concentration levels for leachates -1 of the 1st 24 h, and -2 of the 2nd 24 h, were determined using conventional argentometric titration with silver nitrate and potassium chromate as indicator.

3.1.7 Preparation and Analysis of Set Product Leachate for Preliminary Assessment of TOC Leaching Levels at Ambient Temperature

Duplicates of the set product were soaked in carbon-free distilled water and allowed to slowly swirl for 24 hrs at laboratory ambient conditions. The aqueous phase was similarly stirred and filtered, as before, and the filtrate was analyzed for TOC on a Schimadzu 500 TOC Analyzer.

3.1.8 Determination of TOC Leaching Levels at Higher-than-Ambient Temperature for Differing Leaching Times in Presence and Absence of Chlorine Residual

Three sets of duplicates of the hard-set product were soaked in carbon-free distilled water. One set was allowed to leach for ca. 6 hrs, and the other two for ca. 24 hrs, all at temperatures of ca. 45 °C ($45 \pm 3^\circ\text{C}$). The aqueous phase of one of the 24-hrs duplicate leaching sets was carefully decanted. All these sets of replicates were divided into two groups. One group was treated with the appropriate amount of chlorine enough to ensure a free residual of 0.5 to 1 ppm, while the other group was left unchlorinated. TOC levels for all of these replicates were similarly determined on Schimadzu 5000 TOC Analyzer.

3.1.9 Determination of Base/Neutral and Acid Extractable Organics in Leachates at Ambient and Higher Temperatures for Variable Leaching Times with and without Residual Chlorine

Two sets of duplicates cubicles of hard-set X-Pando product were soaked in a liter of DW as before, and allowed to leach for about 72 hrs at ambient temperature. One set was treated with the appropriate amount of chlorine enough to ensure a free residual of 0.5 to 1 ppm. The other set was left without chlorine. Both samples were subjected to Base/Neutral and Acid Extractable organics on the basis of the EPA method # 625. The extracts were qualitatively analyzed using GC/MS techniques.

A similar set of leaching experiments, also in presence and absence of the same level of chlorine residual, were carried out at higher temp. of ca. 45 ± 3 °C. One set of duplicates were allowed to leach for 24 hrs, whereas the other leaching duplicates were quenched after only 6 hours. Both sets of replicates were extracted and similarly analyzed, as above, for Base/Neutral and Acid Extractable Organics following the same USEPA method # 625

3.1.10 The Starch Test

A simple qualitative laboratory test, the starch test, was applied for both of the 24-hrs ambient temperature leachate and the 24-hrs high temperature leachate for comparison. This was done by adding few drops of an I₂/KI solution to a small aliquot of each leachate solution and noting color development.

3.1.11 EDAX Analysis

A sample of the original powder was EDAX probed for a preliminary qualitative/semi-quantitative screening of elemental constitution of the sample.

3.1.12 Blanks

A reagent blank was prepared and carried throughout all the steps of the above-mentioned analytical procedures.

4. RESULTS AND DISCUSSION

4.1 Original Product Sample

Since most of the deterioration observed along the pipeline was in close proximity to sealed joints, where the applied product showed distinct disbondment, Figs 1 & 2, it was suspected that the disintegrating sealant may release some potentially toxic ingredients into the transmitted potable water. Thus, metal and organic context of the product, as received, were evaluated at the outset.

Metal contents of the original sample powder as screened by EDAX are listed in Fig.(3), together with a tabular listing of the corresponding spectrum of electronic shell transitions. None of the toxic elements stated above were detectable by EDAX analysis. ICPES showed variable levels of Ni, Cu & Zn in the solid sample, with Cd being non-detectable. More rigorous results were presumably obtained from AAS, which again confirmed the absence of Cd while showed Ni content to be the highest of those metals (~ 100 ppm), followed by Zn, Cu & Pb at ~ 11, 10 & 1 ppm respectively.

The original product is a very fine ash-like powder, grey in color, with a finite solubility in water. The soluble portion sustains fine grey particulates in suspension forming an apparently colloidal solution. This solution on filtration across a 0.2 µm membrane filter gave a filtrate solution of pH 8.7 indicating that it is of an alkaline nature.

4.2 Soluble Product Aqueous Sample

Analysis of the solubilized aqueous sample showed results in the ppb levels with Ni again showing the highest level of 146 ppb, followed by Pb, Zn, Cu & Cr at 64, 22, 20.7 & 1.1 ppb, respectively (Table (1), column #4). Cd, however, showed a minute trace of 3 ppb which was not traced in the original sample. This may be due to usual losses of very low levels of trace elements, which could occur during sample preparation.

4.3 Set- product Toxic Metal Ambient and High Temperature Leachates

Toxic metals in the 24-hr aqueous, room temperature leachate-1, showed fairly low trace levels. Ni, Pb & Cu appeared at concentrations levels of 1.1, 3.7 & 3.9 ppb respectively, whereas Hg, As, Se, Cd, Cr & Zn were not detectable (Table (2), column #5). Second and subsequent leachates (leachate-2 & -3) were practically devoid of toxic metals concentrations (Table (2),, columns # 6 & 7).

The high temperature (ca. 45 ± 3 °C) longer time (24 hrs) leaching aqueous solution, though showed a relative increase for some of the toxic metals as compared to the leachates of 6 hr high, and the 24 hrs ambient temp, their absolute metal concentration

values still remain at fairly low levels (Table (3), columns 4 & 5). Cd, which was neither detectable in ambient 24 h leach nor in high 6 h leach, showed a very low trace of 0.5 ppb Table (2),. Cu and Ni showed levels of 6.6 & 8.3 which are roughly double their levels of 3.1 & 4.5 in ambient temperature 24-hr leach.

The high temperature (ca. 45 ± 3 °C) shorter time (6 hrs) aqueous leaching solution also showed fairly low levels of toxic metals. Concentration levels of Cd, Ni, Zn, As, Hg, Se, & Cr showed similar results of non-detectable (or comparable low levels) for both of the 6 hr high temp leachate and the 24 hr ambient temperature (leachate-1). Ni showed a relatively higher level (3.1 vs 1.1), whereas Pb showed a relatively lower level (3.9 vs ND), in the 6 hrs high versus the 24 hrs low, temperature leach, (Table (4), column 4 & 5).

4.4 Chloride Ion Content in Hard-Set Product Leachate

Apart from toxic elements, other elements that had been analyzed and exhibited relatively high concentrations in the leachate include chloride ion. Its content in the leachate of the 1st day (leachate-1) was very high, reaching a value of 574 mg/L; whereas in the 2nd day aq. Replacement solution (leachate-2), it dropped to less than half this value giving a concentration level of 230 mg/L, which is still quite high.

4.5 TOC Sources in X-Pando Product

According to the manufacturer documentations (copy enclosed), there are only two possible sources of organic carbon in the product. These are graphite and starch gum. Graphite is a highly refractory allotrope of carbon, can stand temperatures approaching three thousands °C high, and it is for this reason that is, for example, used as a furnace tube for electro-thermal excitation in atomic absorption spectroscopy, AAS-GTA. In water and oil it only forms insoluble suspensions. It is also resistant to oxidants (chlorine or otherwise) [1]. Thus, the possibility of graphite contribution to the X-Pando aqueous leach of 2.5 ppm TOC obviously doesn't practically exist whether in presence or absence of chlorine.

The only other TOC source remaining is starch gum. This is a polysaccharide carbohydrate, known as dextrin, having the formula $(C_6H_{10}O_5)_n$ where n, the number of

polymer repeating units, can reach hundreds or even thousands. Thus, their molecular weights (MW) are high, ranging from 50 000 to 100 000 or more. They are highly polar macromolecules with extensive inter- and intra-molecular hydrogen bonding. Therefore, being as such, these polar macromolecules can neither be expected to extract into organic solvents, nor to be amenable to GC/MS analysis. As the rest of carbohydrates, these molecules have been shown to play negligible or practically no role as precursors for chlorination disinfection by-products [2]. Thus their potential of forming non-purgable organic DBPs' is also negligible or practically doesn't exist.

4.6 Set-Product Ambient Temperature Leaching and Suspended Particulate Solid Release

Determination of the amount of suspended particulate matter (SPM) released from the hardened cubic paste of X-Pando product which was immersed in a liter of DDI water for the first 24 hrs, the filtered residue gave a value of SPM content of 5.0 mg/L. Replacing the water by a fresh one liter DDI and allowing to stand for another 24 hrs, the filtered residue gave the same value of SPM content of 5.0 mg/L. Changing the DDI water by a 3d liter, and allowing to stand this time for 48 hrs, resulted in SPM content of 3.6 mg/L.

Physical inspection of the soaked sample also revealed visually conspicuous deposits of solid material settling at the bottom of the beaker, particularly at the immediate vicinity of hardened cubic lump. However SPM were determined only in the supernatant aqueous liquor. The repeating value of 5.0 mg/L in a pair of duplicates over two consecutive days, appears to be a saturation value for SPM over 24 hrs. The 3rd leach which was allowed to continue over 48 hrs gave an SPM value of 3.6 mg/L. The reduction in the latter value from 5.0 to 3.6 ppm is likely to be due to that the extended time had allowed more of the relatively heavier particulates to settle.

Physical inspection also revealed several adjacent pores, deep pits (or even cracks) in each of the four tested hardened replicates, Figs. (4) a, b, & c). This, together with the release of suspended and settled particulates to aqueous solution, indicate clearly that the hardened X-Pando material starts eroding, and continue to do, when comes into contact with water.

4.7 Set-Product High Temperature Leaching and Suspended Particulate Matter Release

High temperature leaching of duplicate cubicles in a liter of DDI at 45 ± 3 °C for 24 hrs gave practically no suspended particulate. The values obtained (~ 0.004 ppm) were within the experimental error under the conditions of the determination. This is quite an interesting result with respect to ambient temperature which showed finite, and fairly high SPM load. The most plausible rationale of this peculiar behavior is that the SPM leaching load obtained at room temperature was composed mostly of starch particulates in small microscopic starch granules, with diameters exceeding the $0.2 \mu\text{m}$ membrane filter pores and thus are largely trapped by the filter. At warmer temperatures these molecules leach as dissolved starch molecules rather than in micro-granular form.

It is also very interesting to note that the distinctly conspicuous black color of the residue obtained at room temperature vacuum filtration of the leachate, completely disappeared in case of the higher temperature leachate, resulting in a distinctly clear membrane filters Fig. (5). It is also worthwhile to note that the filtration of the leachate was not carried out while hot but, rather, after quite a while of standing sufficient to bring it to room temperature. Thus, apparently, the process of granular particulate dissolution is an irreversible phase transition process. The scanning electromicroscope (SEM) photographs also support this observations (Fig. 6).

4.8 Set-product TOC Content in Ambient Temperature Leachates

The organic carbon-free leachate of the hardened product showed a fairly low TOC content of 2.5 ppm.

4.9 Base/Neutral and Acid Extractable Organics in Leachates at Ambient and Higher Temperatures for Variable Leaching Times with and without Residual Chlorine

The suggested “Base/Neutrals and Acid Extractable Organics” is a procedure well suited to environmental aqueous samples containing a myriad of various unknown organic Compounds, particularly those containing petroleum spill contaminants [3].

It is not equally suitable for an industrial sample with a well-defined source of organic carbon, as it is the case of X-Pando special product, containing starch gum (dextrin) as the only source for aqueous leaching of TOC. In case of the claim of running the experiment in presence of residual chlorine, the obvious presumption here is that chlorine oxidizes the available TOC as a precursor for formation of chlorinated disinfectant by-products (DBPs). Such an assumption is unfounded in X-Pando case, since, unlike humic substance, carbohydrate (including starch gums) have not been reported in the literature as DBPs' precursors. On the contrary carbohydrates (including Dextran & several other carbohydrates), have been shown to have negligible or no contribution to chlorinated DBPs formation potential. In an experiment which reacted 10 ppm of chlorine with 10 ppm of each of several carbohydrates as well as a model humic acid for 72 hrs, the result was that each of the hydrocarbons (including Dextran) recorded a concentration of less than 1 ppb, compared to the humic acid, which recorded 92 ppb. The conclusion was that carbohydrate have negligible contribution to DBPs formation potential. However, in order to practically verify these inferences, it was decided to carry on with the method and apply it to all leachate samples at different temperatures and different leaching times.

As it was anticipated at the outset, none of the Base\Neutral and Acid Extractable Organic compounds were detectable in any of the leachates whether at ambient or at higher temperatures, for longer or shorter leaching times, with or without residual chlorine (Table 5).

4.10 Set-product TOC Content in High Temperature Leachates

In contrast to low concentration level in ambient temperature set X-Pando leachate, TOC level in high temperature leachate showed a large increase Table (6). This increase may be thought of as an indication of an increased solubility of the product in general at warmer temperatures leading to an added overall dissolution of the product. Also, at both temperatures physical inspection of leached cubes showed comparable degree of erosion as reflected in deep pitting and cracking, Figs. 4 and 7. However, this doesn't seem to be the case since the increase in TOC is quite large (or even inordinate) compared to the corresponding increase in temperature and the cementing nature of the product. Yet, when this increase is read together with the disappearance of suspended

solids in the high temperature leachate, it gives a clearer indication that suspended particulate matter dissolution is more likely to be the cause of elevated TOC level. In addition, if this is the case, it also indicates that this dissolving SPM must be of a largely organic nature. Furthermore, as it has been already deduced above, the main source of TOC can only be the starch component of the product. Thus, If this is truly the case, then a simple laboratory test should definitively verify this hypothesis.

4.11 Starch Test for Low and High TOC Content

Not surprisingly, the *starch test did confirm* that the TOC in the leachates is mainly starch by giving, on the spot, the distinct blue color characteristic of starch-iodine complex. This positive result was obtained only with the high temperature, high TOC sample ~ 58 ppm, whereas with the low TOC content sample of ~2.5 ppm, the color obtain, brownish/yellow, was merely that of the added iodine solution drops (Fig. (8)).

5. CONCLUSIONS

1. Based on the results of the analysis carried out under the conditions specified above, it can be concluded that toxic metals content, viz. : mercury, arsenic selenium, cadmium, lead, nickel, chromium and zinc, in the leaching aqueous solution is low and does not seem to pose an immediate threat under the pipeline massive water flow and dilution conditions . This statement is expected to hold at different environmentally attainable high and low temperatures due to seasonal variations.
2. The hardened material seems to lose coherence easily when it comes into contact with water. This is reflected in the continuous release of suspended particulate matter detritus (SPM) into the aqueous phase. This behavior is likely to be a major cause leading to erosion/corrosion phenomena.
3. The high chloride concentration in the initial and subsequent leaching experiments, points strongly to the possibility of chloride initiated corrosion.
4. The presence of starch in the product seems to be another major cause of its erosion under both cold and warm environmental temperatures. This is apparent from the sizable increase in dissolved total organic carbon at higher temperatures

which, certainly leads to loss of integrity of the product by depletion of one of its carbonaceous components.

5. At low temperatures, however, starch seems to be an even more serious cause of erosion of the product, since it is released in form of particulates starch granules, which appear to strip out with it significant amounts of fine graphite particulates. Thus, at cooler temperatures the presence of starch leads to the depletion of two components, viz. starch and graphite, which represent the two sources of organic carbon in the product; whereas in warmer temperatures only one component, which is starch itself, is significantly depleted. This phenomenon of simultaneous starch and graphite removal was demonstrated in the conspicuous black color of the residue of ambient temperate leachate on the membrane filter, which totally disappears leaving a clear and colorless membrane filter in case of warm temperature product leaching.
6. The above observation is consistent with the low values of dissolved TOC and high SPM obtained at ambient temperature; contrasting a high dissolved TOC and low SPM at elevated temperature. The starch at ambient temperature was depleted mostly as filterable particulates which contributed together with adsorbed carbon to an elevated SPM and low TOC, whereas the opposite occurred at higher temperature.

6. RECOMMENDATIONS

1. From this case study, it can be seen that comprehensive characterization, though might be more time and effort consuming, yet it can be helpful in avoiding some costly consequences. This is particularly true in case of those products which are of constructional or repetitive application nature. Thus, it is recommendable that products such as sealants, paints, lining materials, etc., be subjected to somewhat detailed initial evaluation studies.
2. The above evaluation studies may also encompass product persistence and integrity under prevailing environmental interactions as well as product stability versus degradability and shelf life.

7. REFERENCES

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Table 1. Analysis of Soluble And Leachable Aqueous Parts :

| S.No. | Metals | Units | Soluble Parts | Leachate-1 | Leachate-2 | Leachate-3 |
|-------|--------|-------|---------------|------------|------------|------------|
| 1 | Cd | ppb | 3.0 | ND | --- | -- |
| 2 | Cu | -do- | 20.7 | 1.1 | 1.0 | ND |
| 3 | Ni | -do- | 146.3 | 3.7 | ND | ND |
| 4 | Pb | -do- | 64.0 | 3.9 | 1.0 | ND |
| 5 | Zn | -do- | 22.0 | ND | --- | --- |
| 6 | As | -do- | ND | ND | --- | ---- |
| 7 | Hg | -do- | ND | ND | --- | --- |
| 8 | Se | -do- | ND | ND | --- | --- |
| 9 | Mg | -do- | 2340 | 120 | 90.0 | 80.0 |
| 10 | Na | -do- | 65.6 | 12.5 | 2.0 | 2.0 |
| 11 | Cr | -do- | 0.14 | ND | --- | ---- |

Table 2. Analysis of Solid

| Metals | %Wt | ppm |
|--|---------------------|--------------------|
| Cadmium | ND | ND |
| Copper | 0.001% | 9.89 |
| Lead | 0.00016% | 1.60 |
| Nickel | 0.0100% | 101.6 |
| Zinc | 0.0011 % | 11.38 |
| | Leachate-1 | Leachate-2 |
| Chloride Conc _n (Argentometric) | 574 ppm (0.057%) | 230ppm (0.023%) |

Remarks:

- i. Reagent blanks were also run and subtracted from the corresponding values.
- ii. Analyses shown in tables (1) & (2) were done by AAS (GBC & P. Elmer), except Cl⁻

Table 3. Analysis of Aqueous Leachate at Room Temp & 45°C for 24 hrs:

| S.No. | Metals | Units | Leachate-1 (After 24 Hours at Room Temperature) | Leachate (After 24 Hours At 45°C) |
|-------|--------|-------|---|-----------------------------------|
| 1 | Cd | ppb | ND | 0.5 |
| 2 | Cu | ppb | 1.1 | 6.6 |
| 3 | Ni | ppb | 3.7 | 8.3 |
| 4 | Pb | ppb | 3.9 | 2.4 |
| 5 | Zn | ppb | ND | ND |
| 6 | As | ppb | ND | ND |
| 7 | Hg | ppb | ND | ND |
| 8 | Se | ppb | ND | ND |
| 9 | Cr | ppb | ND | ND |
| 10 | Mg | ppm | 120.0 | 168.0 |
| 11 | Na | ppm | 13.0 | 18.0 |

Remarks:

- i. Reagent blanks were also run and subtracted from the corresponding values.
- ii. Analyses were done by AAS (GBC & Perkin Elmer).

Table 4. Analysis of Leachable Aqueous Parts at 45 ± 3 ° C For Different Time Periodsn (6 & 24 hrs)

| S.No. | Metals | Units | Lecheate After 6Hours (45 ⁰ C) | Lecheate After 24 Hours (45 ⁰ C) |
|-------|--------|-------|---|---|
| 1 | Cd | ppb | ND | 0.5 |
| 2 | Cu | ppb | 3.1 | 6.6 |
| 3 | Ni | ppb | 4.5 | 8.3 |
| 4 | Pb | ppb | ND | 2.4 |
| 5 | Zn | ppb | ND | ND |
| 6 | As | ppb | ND | ND |
| 7 | Hg | ppb | ND | ND |
| 8 | Se | ppb | ND | ND |
| 9 | Cr | ppb | ND | ND |
| 10 | Mg | ppm | 55.0 | 168.0 |
| 11 | Na | ppm | 9.0 | 18.0 |

Remarks:

- i. Reagent blanks were also run and subtracted from the corresponding values.
- ii. Analyses were done by AAS (GBC & Perkin Elmer).

Table 5. Organic Priority Pollutants EPA # 625

| S.No. | Sample particulars | Result |
|-------|---|--------|
| 1 | Xpando, at room temperature for 3 days without chlorine | ND |
| 2 | Xpando, at room temperature for 3 days with chlorine | ND |
| 3 | Xpando, at 45 deg C for 6hours without chlorine | ND |
| 4 | Xpando, at 45 deg C for 6 hours with chlorine | ND |
| 5 | Xpando, at 45 deg C for 24hours without chlorine | ND |
| 6 | Xpando, at 45 deg C for 24 hours with chlorine | ND |

ND : Not Detected

Note: Residual chlorine of about 0.5 to 1 ppm approximately was maintained in samples with chlorine.

Table 6. TOC for X-Pando leachate for 6 and 24 Hrs with and without Chlorination

| Sample ID | Description | TOC mg/L | pH |
|-------------------|---------------------------------|-----------------|-----------|
| S-1 dated 22/6/03 | With chlorine residual 6Hrs | 23 | -- |
| S-2 dated 22/6/03 | Without Chlorine 6Hrs | 23 | -- |
| S-3 dated 23/6/03 | With Chlorine residual 24Hrs | 58 | 7.7 |
| S-4 dated 23/6/03 | Without Chlorine residual 24Hrs | 54 | 7.7 |

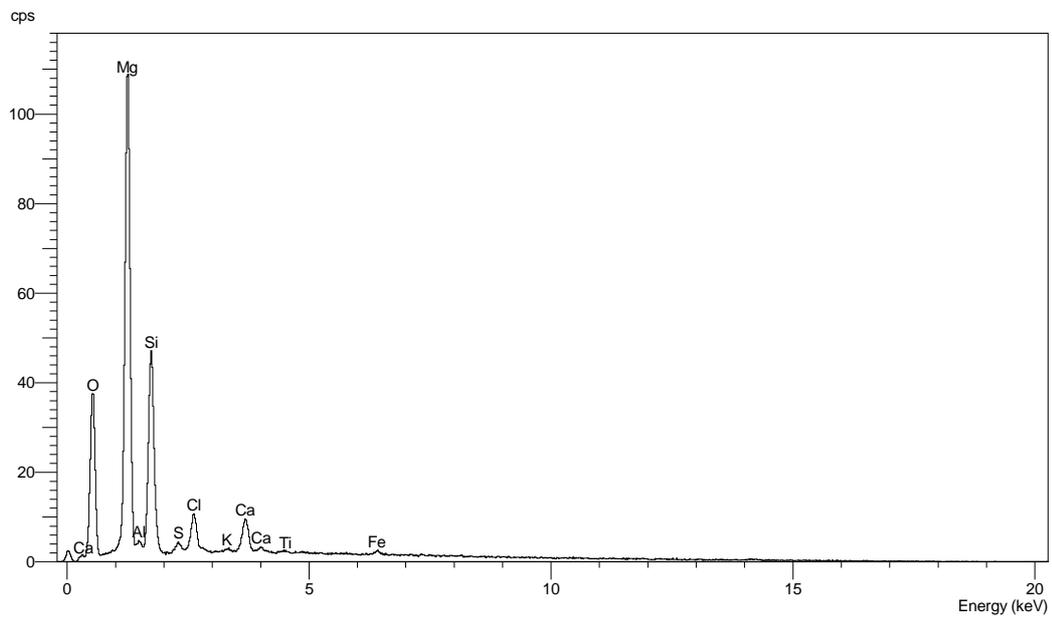


Figure 1*. Disbondment of X-Pando sealant



Figure 2*. Pipe sample showing corroded steel and cement surface below X-Pando sealant

* from Ref. [1]



| Elmt | Spect. Type | Element % | Atomic % |
|-------|-------------|-----------|----------|
| O | K ED | 42.40 | 55.27 |
| Mg | K ED | 31.95 | 27.41 |
| Al | K ED | 1.60 | 1.23 |
| Si | K ED | 15.41 | 11.44 |
| S | K ED | 0.70 | 0.46 |
| Cl | K ED | 3.16 | 1.86 |
| K | K ED | 0.37 | 0.20 |
| Ca | K ED | 3.25 | 1.69 |
| Ti | K ED | 0.28 | 0.12 |
| Fe | K ED | 0.88 | 0.33 |
| Total | | 100.00 | 100.00 |

Figure 3. Analysis of Soluble and Leachable Aqueous Parts



Fig. 4 (a) General experimental layout (ambient)

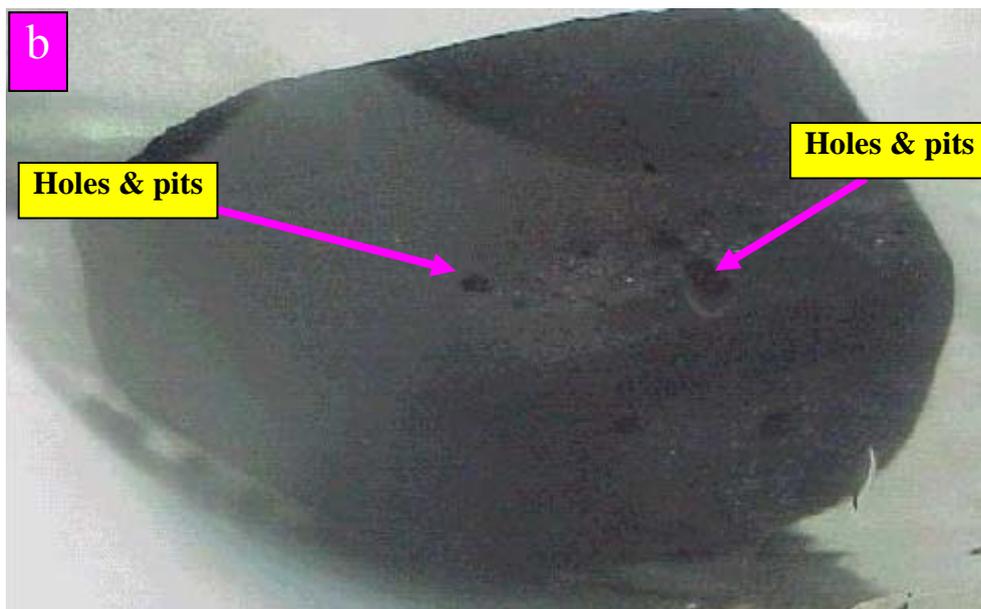


Fig. 4. (b) Close-up of replicate cube-like hardened material showing holes, and pits in the set material (ambient)

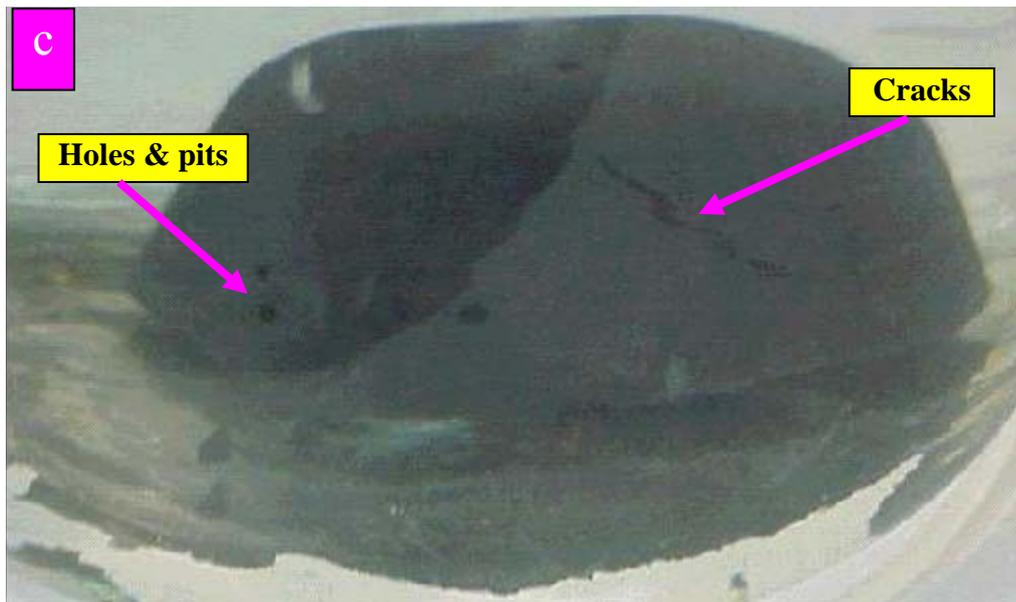


Fig. 4 (c). Close-up of replicate cube-like hardened material showing holes, pits and cracks in the set material (ambient)

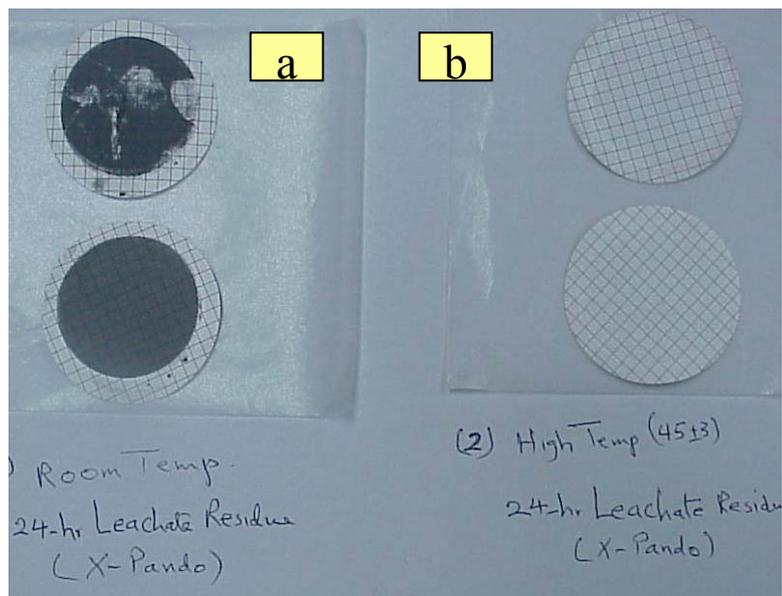


Figure 5. Filtration Residue of X-Pando SPM after 24 h leaching (Filters, Pair (a) Room Temp. black residue, Pair (b) High temp. colorless residue)

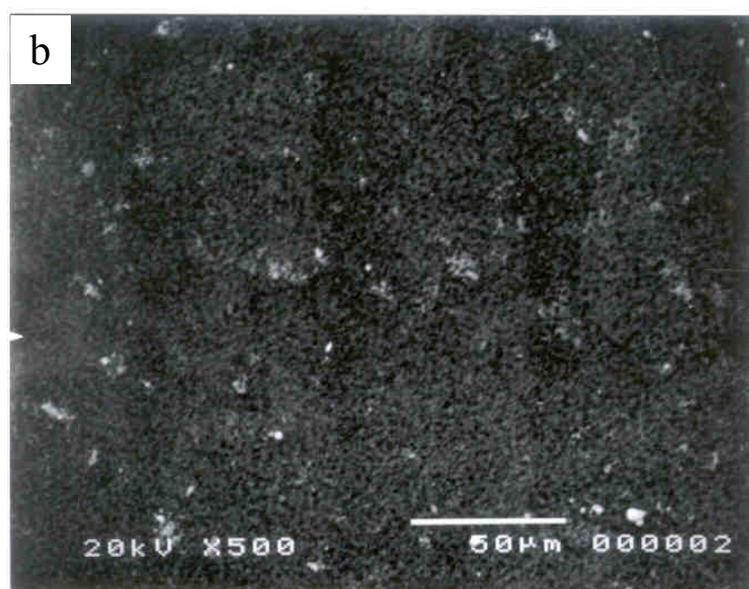
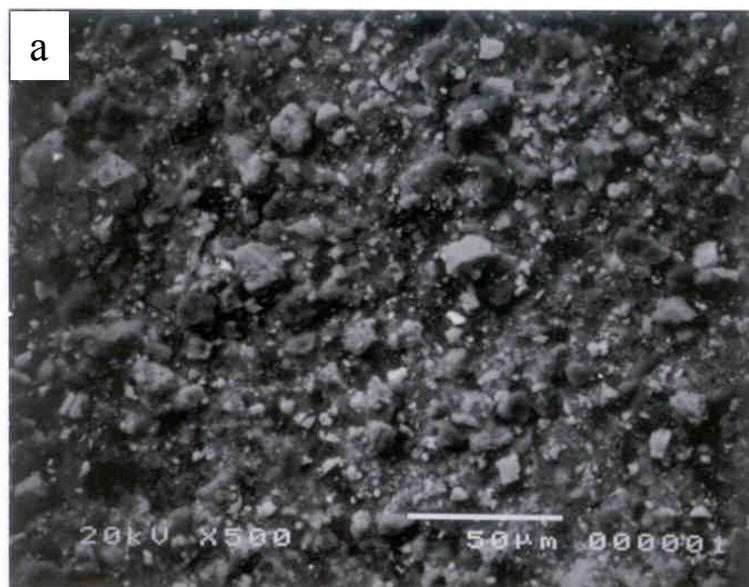


Figure 6. SEM of Filtration Residue of X-Pando SPM after 24 h leaching (a) Room Temperature (b) High Temperature

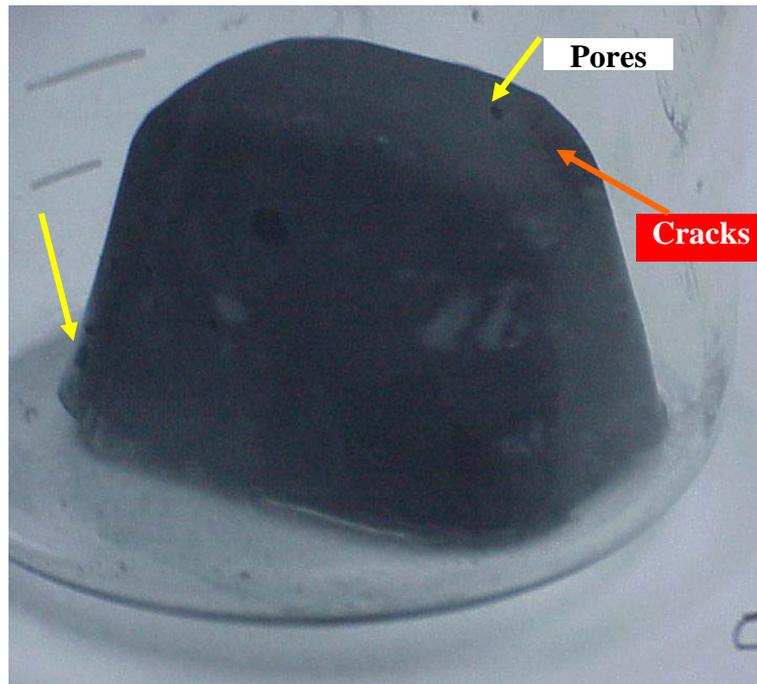


Figure 7. Hard-set X-Pando after 6 hrs leaching in aqueous solution at 45°C

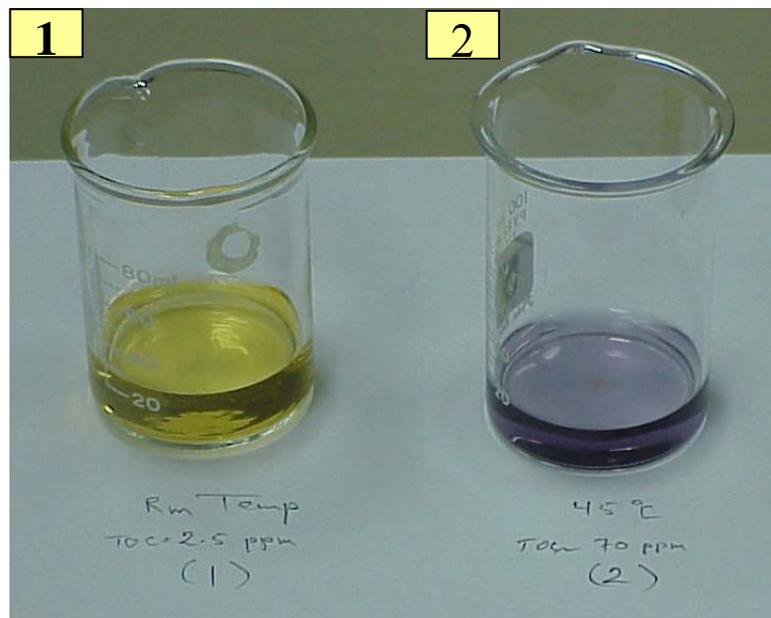


Figure 8. Starch test for: Beakers (1) Left 2.5 ppm TOC content and (2) Right 58 ppm TOC content