

# Latest Findings in In-situ Remediation of Hydrocarbon Impacted Soils using Hydrogen Peroxide

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## Abstract

There has been a concerted effort over the last few years in Alberta to increase our level of understanding of hydrogen peroxide ( $H_2O_2$ ) technology as an oxidation remediation technique for hydrocarbon-contaminated sites.  $H_2O_2$  (and Fenton's reagent - a mixture of  $H_2O_2$  and iron salts) oxidizes contaminant hydrocarbons, and it degrades and releases oxygen and bio-stimulates the subsoils leading to a faster aerobic degradation. This paper gives an overview of the  $H_2O_2$  technology and describes a successful field application that has subsequently led to a number of research projects at the University of Calgary. The field application proved economical and was successful in a restricted access situation (Mahmoud et al, 2000, 2003). Two research projects have been conducted since the completion of the field application. The first focused on examining the potential of  $H_2O_2$  in causing volume change in hydrocarbon-contaminated silty sands (Mahmoud et al, 2003; Mohamed et al, 2002). The second has concentrated on examining the generation of gases leading to soil volume change, as well as determining the optimal volume of Fenton's reagent required for remediating diesel-impacted soil. This latest study has also examined the zone of influence of  $H_2O_2$  as it is being injected in a column of soil.

In the field program,  $H_2O_2$  was injected to destroy contaminants extending underneath a building floor slab and adjacent to two foundation piles where contaminants included volatile organic compounds (xylenes), total petroleum hydrocarbons (both volatile, TVH and extractables, TEH), and isopropyl alcohol. While measurements using tilt-plates indicated little or no ground movement during and after  $H_2O_2$  injection, it was considered prudent to study the effect of  $H_2O_2$  treatment on soil volume change under (controlled) laboratory conditions. The volume change experiments indicated an almost instantaneous settlement in diesel-impacted silty sand treated with  $H_2O_2$  concentrations less than 25%. Samples treated with increasingly higher concentrations of  $H_2O_2$  showed a subsequent rebound after the initial instantaneous settlement. With increasing  $H_2O_2$  concentration the rebound of samples led to heave as the  $H_2O_2$  concentration increased beyond 25%. This behaviour was explained in terms of gas production, decomposition of organic matter, and rearrangement of soil particles.

In order to determine the mechanism leading to soil volume change, a follow-up laboratory investigation was undertaken. The optimal  $H_2O_2$  concentration was evaluated for

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remediation of diesel-impacted soils. About 65% of diesel range organics was removed when 5 grams of a sandy silt was treated with 20 mL of 20% H<sub>2</sub>O<sub>2</sub>. Gas production during the reactions led to a substantial decrease in hydraulic conductivity of samples. H<sub>2</sub>O<sub>2</sub> injection experiments yielded a cone of treated soil around the point of injection.

## 1. Introduction

Petroleum hydrocarbon-induced contamination in soil has become a widespread problem, due mainly to leaks from underground storage tanks and spills from either pipeline ruptures or tank rail derailments (US EPA, 2000; Lyman et al, 1990). Hydrocarbon spills can be a source of contamination of ground surface, surface aquifers and groundwater supplies, as well as fire and explosive hazards through vapour migration and accumulation. Established remediation technologies for hydrocarbon-contaminated soils include contaminant removal, bio-remediation, thermal treatment, soil washing and soil flushing. Advanced oxidation process (AOP), a chemical treatment method, is one of the technologies that has shown promise in destroying and degrading hazardous wastes in water and soils using various oxidants. The most widely documented application of AOP to date relates to the use of Fenton's reagent, a mixture of hydrogen peroxide and iron salts. AOP can offer certain advantages over the other remediation technologies, including

- easy operation, low cost both in chemicals and labour, and short treatment time;
- readily delivered to deep subsurface and/or locations with restricted access (Mahmoud et al, 2000, 2003) and
- source contaminants elimination rather than removal.

Documented cases of using H<sub>2</sub>O<sub>2</sub> to successfully oxidize petroleum hydrocarbons include Mahmoud et al, 2000, 2003; Spencer et al, 1996; Pardiek et al, 1992; Watts, 1992; and Watts et al, 1991. The hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) injection process has been used to remediate silty clay soils at approximately 20 sites in Western Canada (Mahmoud et al, 2003). The majority of sites have been former fuel facilities where the remediation criteria used were the 1994 Alberta Environmental Protection (AEP) guidelines for petroleum storage tank (PST) sites (AEP, 1994). Numerous sites have also been remediated in the United States using this technology. Significant reductions (in the order of 50 to 95%) have been observed in petroleum hydrocarbon concentrations following H<sub>2</sub>O<sub>2</sub> injections in lacustrine and clay till soils, while H<sub>2</sub>O<sub>2</sub> injections are difficult to complete in soils with significant proportions of gravel or cobbles (Nichols Environmental (Canada) Ltd, 2001).

## 2. Hydrogen Peroxide Technology

While hydrogen peroxide is in itself an oxidant, the presence of iron catalysts leads to the decomposition of hydrogen peroxide by ferrous ion (Fe<sup>2+</sup>) to form hydroxyl radicals (OH·). The oxidant degrades organic contaminants upon contact. The reaction is non-selective, and the organic molecules are oxidized in no particular sequence (Watts, 1992). This contrasts to biological degradation where the more easily degraded compounds tend to be degraded first, leaving progressively less degradable contaminants as residuals (Pardiek et al, 1992). A

disadvantage of the non-selective nature of the reaction is that the oxidant will react with soil organic matter, thus increasing the volume of oxidant required. The amount of hydrogen peroxide that is required for successful chemical reaction has been found to vary between 2 to 20 times that of the amount of the peroxide necessary stoichiometrically (Nichols Environmental (Canada) Ltd., 2001). The additional hydrogen peroxide is consumed by the natural organic content of the contaminated soil (Chen et al., 1998), carbonates and in scavenging reactions.

As mentioned, hydrogen peroxide, when catalyzed by ferrous ion, decomposes rapidly to produce hydroxyl radicals, OH·. Haber and Weiss (1934) proposed the following equation to describe that reaction:



Hydroxyl radicals are capable of oxidizing most organic compounds (Walling, 1975). As for oxidation of hydrocarbons, the mechanism is not well understood but is usually associated with the following two pathways:

1. oxygen addition - where hydroxyl radical adds to an unsaturated organic compound (alkenes or aromatic rings) to first form a free radical that eventually transforms to organic product (see Equation 2 below); and
2. hydrogen abstraction - in which a free radical and water are initially formed when hydroxyl radical reacts with saturated compounds (Equation 3), followed by further reactions depending on oxidant concentration and pH (Bishop et al., 1968; Sedlak and Andren, 1991; Watts and Dilly, 1996; Nesheiwat and Swanson, 2000).



The decomposition is referred to as Fenton's reaction and it occurs through a set of primary reactions, including the ones given below (Walling, 1975):



The injection of H<sub>2</sub>O<sub>2</sub> treats both saturated and unsaturated soils, as well as groundwater. Commercially procured H<sub>2</sub>O<sub>2</sub> is diluted with water during the injection process. Dilution decreases the rate of reaction while increasing the soil moisture content. It also allows the concentration of oxidant to be varied during the injection process. The initial concentration is pre-determined based on contaminant type and concentration. The reaction is exothermic, which can spray mud and water (steam) out of the injection points. The system operator can reduce the

oxidant concentration for safety reasons if violent reactions are anticipated. Typically, injections of H<sub>2</sub>O<sub>2</sub> are conducted at concentrations in the range 10%-25%.

### **3. In-Situ Treatment using Hydrogen Peroxide**

The case study that was reported by Mahmoud et al (2000, 2003) described a packaging material production plant site where there had been leakage from an underground storage tank (UST) that was intended for the purpose of collecting spills or overflows from a chemical storage room. Physical removal of the UST and adjacent contaminated soils had not resulted in the complete removal of the contaminants. Residual contamination remained underneath a warehouse building and around two foundation piles that supported the building superstructure. The building owner required remediation of the residual contaminants before it would release the tenant from its obligations. The tenant agreed to bring the site into compliance with the applicable regulatory criteria by removing (or degrading) residual hydrocarbons in soils. The residual contaminants included VOC, TPH and isopropyl alcohol. The remediation of the residual contaminants was completed using H<sub>2</sub>O<sub>2</sub> technology.

Subsurface conditions consisted of silty clay and clayey silt to depths of 6 m below the surface, with sand fill encountered beneath the warehouse floor slab.

Confirmation samples that exceeded the Alberta Tier I criteria – adopted as the first screening level - were subsequently screened against the interim CCME criteria – the screening level for commercial/industrial land use. This was considered appropriate since (the more stringent) Alberta Tier I criteria could not reasonably be met at all locations on the property, particularly under the existing warehouse building. The PST Level II criteria – adopted as screening for heavier petroleum hydrocarbon products – were used to address residual concentrations of TEH in soil (TEH is not included in the Alberta Tier I or CCME criteria). The Level II criteria for fine-grained soils applied except for any contamination in the sand fill beneath the warehouse floor slab, to which the criteria for coarse-grained soils applied.

The treatment involved advancing treatment points at approximately 0.6 m centres in the contaminated area and beneath the floor slab in the former chemical storage room. Two applications of H<sub>2</sub>O<sub>2</sub> were injected, the first was approximately 2000 L of 16% H<sub>2</sub>O<sub>2</sub> and the second was approximately 1600 L of 20% H<sub>2</sub>O<sub>2</sub>. Tilt-plates – installed for monitoring purposes during and after H<sub>2</sub>O<sub>2</sub> treatment – showed little or no ground movement.

## **4. U of C Laboratory Program**

### **4.1 Materials and Methods**

The laboratory investigations were conducted on spiked diesel-contaminated soils. Diesel has a carbon number predominantly in the range C<sub>8</sub> to C<sub>25</sub> (Weisman, 1998), and a density of about 0.84 g/mL (Hwang, 1999). No. 2 diesel was used as target contaminant and was obtained from a local Calgary gas station. The soil volume change experiments were conducted on clayey silty fine sand whereas subsequent laboratory studies utilised a sandy silt collected from the U of C grounds. Ottawa sand was used as control material.

The volume change experiments were conducted using fixed ring consolidometers. Digital pressure gauges and electronic transducers were used and data-logging was with a computer. Clean soil was spiked with diesel fuel to concentrations of 2000 and 5000 mg/kg by dissolving the diesel in 150 ml of di-chloromethane (DCM) and mixing thoroughly. The DCM was then evaporated. The spiked soil specimens were compacted to maximum dry unit weight of 16.4 kN/m<sup>3</sup> – corresponding to optimum moisture content of 16% – using the standard Proctor method. A constant pressure of 30 kPa was applied, and the specimens were allowed to consolidate for 24 hours prior to inundation.

Vial tests as well as bench-scale injection and infiltration tests were also conducted. To evaluate the optimal amount of H<sub>2</sub>O<sub>2</sub> per gram of diesel-impacted soil, soils spiked with diesel of 5000 mg/kg were treated in vial tests by H<sub>2</sub>O<sub>2</sub> of varying concentrations (5, 10 and 20% by weight) and volumes (5, 10, 20, 40 and 60 mL). Other factors such as iron concentration and oxygen generation were also examined. For vial tests, 5 g of soil was placed in vials followed by the addition of Fenton's reagent. The reaction was allowed to proceed for 30 minutes at room temperature before separation by centrifugation. The settled soil was then collected and air-dried under fume hood for 4 hours.

In infiltration and injection tests, the sandy silt was spiked with 5000 mg/kg diesel, premixed with about 10% distilled water, and then compacted using the standard Proctor method. For infiltration tests, H<sub>2</sub>O<sub>2</sub> served as the infiltrating liquid in the permeameter-like column packed with soil sample. For injection tests, an injector was inserted in soil sample compacted in a plexiglass cylinder tube with one end glued onto a plate. H<sub>2</sub>O<sub>2</sub> solution was injected using a 60 mL syringe. Four hours after the treatment a total of 80 soil samples were collected (using thin wall steel tube) at 20 locations from four layers of the 80 mm long soil column. Diesel range organic (DRO) was analyzed on a Hewlett-Packard 6350 gas chromatograph (GC) fitted with flame ionization detector (FID) and an HP autosampler using 2 mL HP GC vials.

Further details of the experimental protocol and the methods followed may be found in Mohamed et al (2002) and Xu (2003).

## **4.2 Results and Discussion**

### **4.2.1 Effect of H<sub>2</sub>O<sub>2</sub> on Soil Volume Change**

The experiments revealed that the use of H<sub>2</sub>O<sub>2</sub> in soil remediation can lead to volumetric change. Below a 15% concentration of H<sub>2</sub>O<sub>2</sub>, treated soils experienced immediate volume reduction; this reduction in volume decreased as the H<sub>2</sub>O<sub>2</sub> concentration increased. Volume reduction was linked to the decomposition of organic matter and consequent re-arrangement of soil particles. At higher H<sub>2</sub>O<sub>2</sub> concentrations (15-25%) soil volume increased – due to gas production – but as the gas pressure dissipated over time, the sample consolidated and the net effect was little or no change in volume. 30% H<sub>2</sub>O<sub>2</sub> concentration applied to 5000 mg/kg diesel-contaminated silty sand caused an increase in void ratio of about 25%. For further details of the results refer to Mahmoud et al (2003) and Mohamed et al (2002).

#### **4.2.2 Iron as Catalyst in Effectiveness of H<sub>2</sub>O<sub>2</sub> Treatment**

Our results showed virtually no H<sub>2</sub>O<sub>2</sub> decomposition when 10 mL of H<sub>2</sub>O<sub>2</sub> was added to Ottawa sand, whereas the addition of either ferrous or ferric ions markedly promoted H<sub>2</sub>O<sub>2</sub> decomposition. Test results also showed insignificant difference in diesel range organics (DRO) degradation of samples amended with soluble iron and those without external amendment (the sandy silt had a natural iron content > 1% by weight). Five mL of 10% H<sub>2</sub>O<sub>2</sub> degraded 34% of DRO without external amendment, whereas the DRO degradation was 35% when the soils were amended with iron of varying concentrations. The results demonstrated that naturally-occurring iron was able to provide sufficient catalytic activity for Fenton's reaction. Similar results have been reported by Xu (2003) for silty clay soils.

The mechanism of mineral iron as an effective catalyst includes dissolution with release of soluble irons and heterogeneous catalysis on mineral surfaces (Tyre et al, 1991). Thus, naturally-occurring iron as catalyst is especially beneficial in complex site conditions where homogeneous external iron addition is difficult.

#### **4.2.3 Oxygen Generation**

About 400 mL of oxygen was generated in experiments where 5 g of spiked (5000 mg/kg diesel) sandy silt was treated with 10 mL of 10% H<sub>2</sub>O<sub>2</sub>. It is considered that the large volume of oxygen that is generated likely affects the soil structure.

#### **4.2.4 Diesel Degradation Efficiency**

Figure 1 presents the percent DRO removals of 5 g of sandy silt treated with varying concentrations and volume of hydrogen peroxide. When using 10% and 20% H<sub>2</sub>O<sub>2</sub>, the percent diesel removals increased with an increase of H<sub>2</sub>O<sub>2</sub> up to 20 mL. Further application of H<sub>2</sub>O<sub>2</sub> by increasing the volume to 40 mL and 60 mL appeared to result in a smaller relative increase in diesel degradation. The highest diesel degradation efficiency (about 70%) was obtained when 60 mL of 20% H<sub>2</sub>O<sub>2</sub> was used.

The application of 20 mL of 10% and 40 mL of 5% H<sub>2</sub>O<sub>2</sub> achieved diesel degradation efficiency of about 57% and 61% respectively. 20 mL of 10% H<sub>2</sub>O<sub>2</sub> and 40 mL of 5% H<sub>2</sub>O<sub>2</sub> (for treating 5 g of soil) correspond to 10 mL of 20% H<sub>2</sub>O<sub>2</sub>; the latter is equivalent to a H<sub>2</sub>O<sub>2</sub> : soil ratio of 2:1 for the 20% concentration. This ratio is different from the 7:1 ratio reported by Nichols Environmental (Canada) Ltd. (2001) based on their field experience with 20% H<sub>2</sub>O<sub>2</sub> applications. The difference may be attributed to the fact that loose soils were used in the laboratory study whereas more compact, undisturbed soils were treated in the field applications.

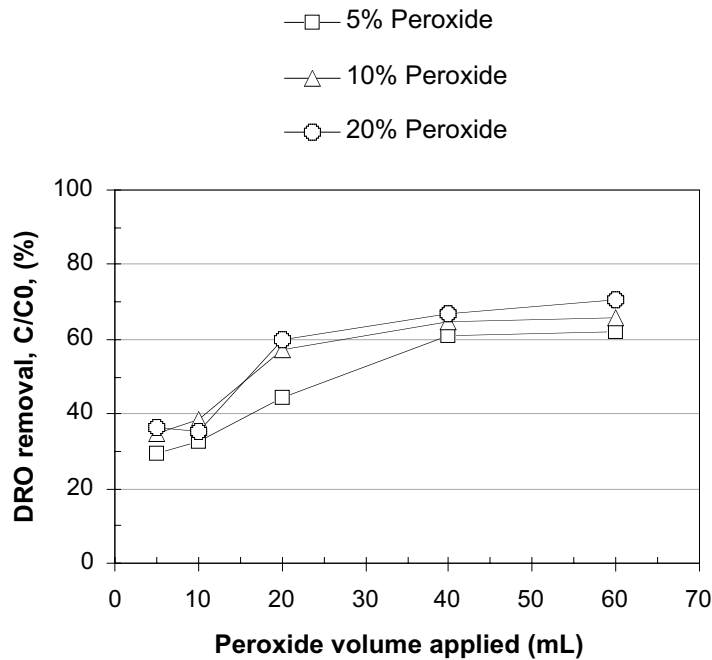


Figure 1: Percent DRO removal as a function of volume of  $H_2O_2$  of varying concentrations for sandy silt (5 g of soil, 5000 mg/kg diesel)

#### 4.2.5 Infiltration Tests

Results of infiltration tests have indicated that  $H_2O_2$  significantly reduces soil hydraulic conductivity as compared to distilled water. It took about 15 minutes for water to pass through the soil column whereas about 3 hours passed before  $H_2O_2$  effluent was observed. It was clear through the side of the plexiglass cell that the downward movement of  $H_2O_2$  was greatly hindered by vigorous gas generation, resulting in the effective pore space being reduced. The hydraulic conductivity for  $H_2O_2$  solution was found to be about 30 times lower than that of distilled water. Thus, it is important to take account of the decreased hydraulic conductivity associated with  $H_2O_2$  reactions within soil.

#### 4.2.6 Zone of Influence in $H_2O_2$ Injection

Figures 2 to 5 show the 3-D plot and contour map for residual DRO concentrations in soil sample after injection of  $H_2O_2$  along the central axis of the sample. The sample was split into 4 horizontal layers. Soil around the central axis was more effectively degraded, resulting in a cone-shaped distribution of diesel degradation efficiency. As much as 30% diesel degradation was achieved near the central axis. The radius of influence was limited as a result of reduced soil permeability and due to the fairly low injection pressure. Minimal diesel degradation was observed along the outer periphery of the soil column.

During the injection, the  $H_2O_2$  moved upward along the injector and exited onto the sample surface. The resulting ‘ponding’ of  $H_2O_2$  covered most of the soil surface around the injector. This phenomenon is similar to the “refusal” that typically occurs in field lime fly ash injection, where the injected liquid often oozes out to the surface. Mahmoud et al. (2003) and Nichols Environmental (Canada) Ltd. (2001) have also reported significant ‘oozing out’ of  $H_2O_2$  in their field applications.

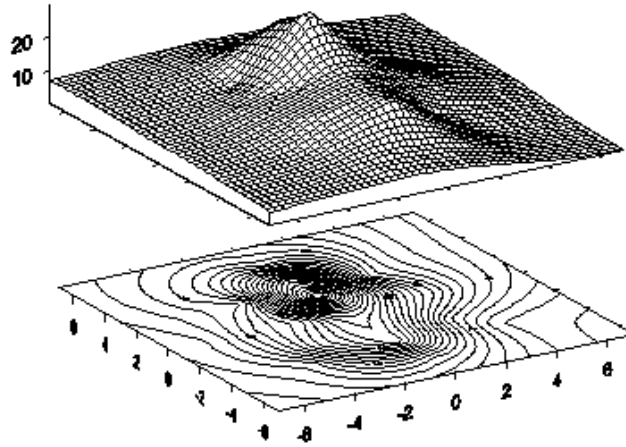


Figure 2: 3-D wireframe plot and contour map for diesel concentration degraded in layer #1 in injection test

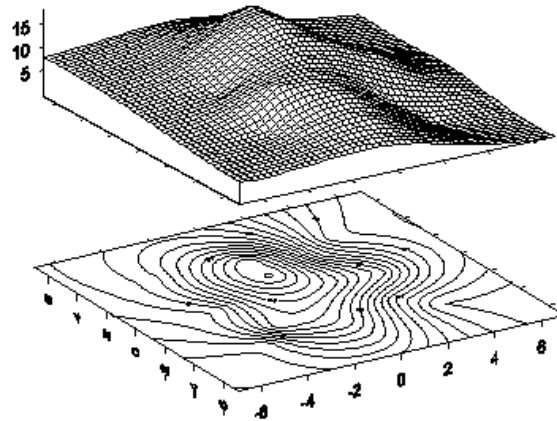


Figure 3: 3-D wireframe plot and contour map for diesel concentration degraded in layer #2 in injection test

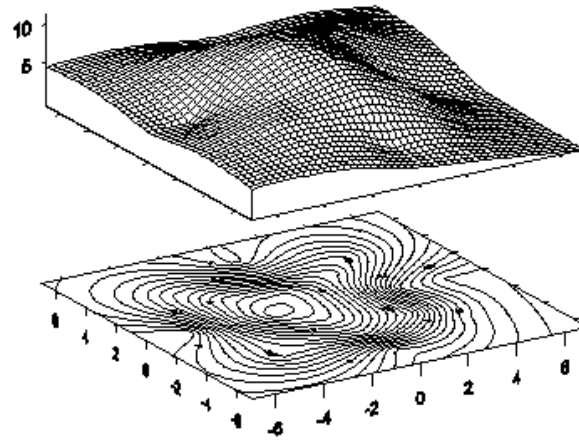


Figure 4: 3-D wireframe plot and contour map for diesel concentration degraded in layer #3 in injection test

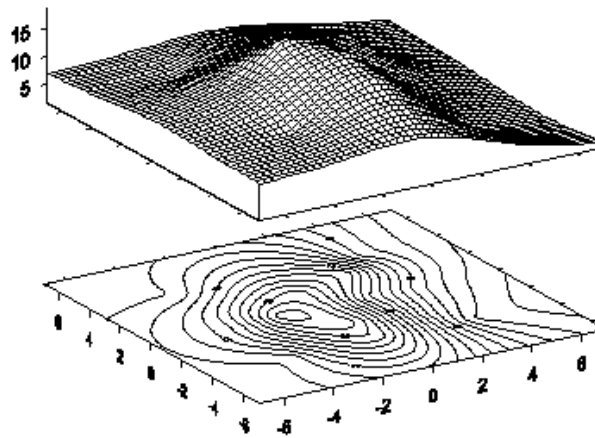


Figure 5: 3-D wireframe plot and contour map for diesel concentration degraded in layer #4 in injection test

## 5. Conclusions

Based on our field and laboratory experience, the following conclusions may be drawn:

- Use of hydrogen peroxide is a viable technology for treatment of hydrocarbon-impacted soils, particularly in areas of restricted access;
- If sufficient iron is present in the soil, external iron amendments to the soils will not have any added benefit;
- Hydrogen peroxide treatment may lead to volumetric change in soil, depending on the concentrations of  $H_2O_2$  and the contaminant;
- The concentration and volume of  $H_2O_2$  were the most significant process variables in Fenton's reaction; high concentration of  $H_2O_2$  increased the diesel degradation efficiency;

- The optimum dosages for remediation of the loose, disturbed sandy silt soil was equivalent to a ratio of about 2:1 (H<sub>2</sub>O<sub>2</sub> : soil) by volume where 20% hydrogen peroxide was used for the treatment;
- Gas generation, specifically oxygen, can be in large quantities during Fenton's reaction, and the large volume of gases generated may lead to a change in soil structure;
- Gas generation may decrease the hydraulic conductivity of soils and this should be considered in estimating the volume of hydrogen peroxide required for treatment of hydrocarbon-contaminated sites; and
- The degradation of hydrocarbons will be highest at locations close to the point of injection.

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