Methods of Water Removal from Quench Oils

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Abstract
This paper describes problems associated with water contamination in quench oils and various methods that can be utilized to remove this contamination. This is water that can be present in the quench oil in use at the furnace, or removing water that is recovered from drag-out and washer solutions. By utilizing the proper water removal methods, companies can reduce their risk of catastrophic consequences due to water contamination. Various methods that can be utilized to remove water from these oils: coalescing, absorptive type media, vacuum distillation, and desorption. A comparison of each method and the advantages and disadvantages of each will be discussed. Additionally, the huge cost savings that can be achieved by removing water from the waste oil stream, allowing oil to be reused instead of processing it as waste, will be examined. The paper will provide case studies and data to show the water removal levels that can be achieved using the proper methods. This paper will demonstrate direct cost savings by reducing water from waste streams, and reducing the risks associated with water during quenching.

Introduction
Water can have a dramatic effect on quenching in oil. The potential for fire, explosion, and injury to people and equipment are ever present and should be taken extremely seriously.

Water can ingress into the oil in various ways, but the most common are: condensation, water-cooled heat exchanger leaks, poor maintenance practices, and washing of parts post heat treat. Some of these can be controlled through best practices, but we should all be aware of the dangers associated with water and proper methods of removing that water, down to an acceptable level.

There are several effective methods to remove water from quench oils. With periodic testing, a level of confidence can be achieved that the oil is in good shape and doesn’t present a risk to your people or equipment. In this paper, the various methods to reduce water contamination in oils will be reviewed. Further, water removal from waste oil streams will be shown to result in large potential cost savings.

Periodic Testing of the Oil
It is extremely important to remember, no matter what your method of water removal, that periodic testing of the oil should be required and those procedures followed diligently [1].

There are two primary methods to measure and control the presence of water in the quench tank. They are as follows:

“Crackle Test” - This is a qualitative, and involves taking a small quantity of oil and heating it up to above the boiling temperature of water. Often a hot plate is used at 320°F (160°C). If bubbling or crackling of the oil occurs, then water is present in the oil [2]. However, the oil will “crackle” only if water is present in quantities above the saturation point (Figure 1). Unfortunately, at this point, it may be too late.

The crackle test is simple and can be done on the shop floor. However, it is not quantitative in nature and will not detect small quantities of water in oil. Water contents greater than 1000 ppm are generally considered to be dangerous, with risk of fire and explosion.

Karl Fischer Test – This is a quantitative test method widely used to determine the quantity of water in oil (ASTM D-6304). This test is based on the Bunsen reaction between iodine and sulfur dioxide. This method is very accurate for used oils, and can determine water content to less than 50 ppm. Measurements are less accurate in new oils, because of interference due to the anti-oxidant additive packages in new oils. It is generally limited to water amounts greater than 2500 ppm of water. Water contents greater than 1000 ppm are generally considered to be dangerous, with risk of fire and explosion.

Figure 1. Bubbling observed during “crackle testing” of oil. Photograph courtesy of Ametek Arizona Instruments, Chandler, AZ.

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Ramifications of Water in Quench Oil

When the water content of the oil is less than the saturation point (approximately 1000 ppm), the water exists as small droplets mixed with the oil. It can also exist as a light emulsification. When the water content is less than the saturation point, it can have some significant effects on the microstructure and distortion of heat-treated parts. The cooling curve of oils containing minute quantities of water show very distinct characteristics [3]:

- Increased stability of the vapor phase;
- Increase length of the vapor phase;
- Increase rate of nucleate boiling; and
- Decreased transition temperature between nucleate-boiling and convection.
- In extreme cases, the transition between nucleate boiling and convection can be completely suppressed.

These changes in the cooling curve behavior of cold quench oil can be observed in Figure 3. It can be observed that the vapor phase becomes significantly more stable, and the maximum cooling rates increase. Also, the temperature at maximum temperature decreases as the water content is increased. At extreme water concentrations the convection phase is suppressed to the approximate boiling temperature of water.

The effect on parts heat treated when water concentration is high, can manifest itself in several ways, including:

- Soft spots from a stable vapor phase;
- Improper microstructure from inadequate quenching;
- Increased distortion and risk of cracking due to the suppression of the transition between nucleate boiling and convection.

Internal investigations have shown that water concentrations as low as 250–400 ppm can result in the formation of non-martensitic transformation products in low hardenability steels.

When the concentration of water exceeds the saturation point (approximately 1000 ppm or 0.1%), it increases the potential for catastrophic fire and explosion. In addition to the hazards to parts indicated above (soft spots, improper microstructure, etc.), there is a severe hazard for fire and explosion. This is due to the generation of large volumes of steam from small quantities of water and from the expansion of steam-laden froth into the work zone of the furnace [4].

Imagine a simple integral quench furnace, with 1000 gallons (3785 liters) of oil. Approximately 1 gallon (3.8 liters) of water (0.1%) is sitting at the bottom of the quench tank because of a leaking air cylinder. The quench tank is being maintained at 170°F (77°C), and a 1000 pound (453 kg) load of parts is quenched from 1600°F. Immediately upon quenching, the bulk temperature of the oil increases approximately 70°F (39°C), resulting in the one gallon of water turning completely to steam. This one-gallon of water creates approximately 215ft³ (6.1 m³) of steam – an expansion of 1600 times. In other words, the 1000-gallon quench tank now contains approximately 1600 gallons of steam. This steam immediately displaces the oil level up, spilling over the furnace hearth. The oil ignites, and expands further. The furnace outer door opens from the pressure, and the froth of the oil-steam mixture is explosively expelled all over the furnace.
the heat treat shop. Once the outer door is thrown a substantial distance, the flame curtain, sensing that the door is not present, ignites, causing the oil/water froth to catch fire. A large fire ensues. Major property damage, and potential loss of life can result (Figure 3 4).

Figure 4. Resulting damage from water in a quench tank.

This simple scenario of a water-oil quench fire unfortunately has been repeated with disastrous results many times in the past several years. The source of the water varies, but has the same catastrophic results. Several heat treat shops have either closed, or experienced long expensive delays while their shop and equipment has been repaired.

The sources of water have often been due to poor maintenance, poor shop keeping, or a lack of understanding of the ramifications of water in the quench oil. In the balance of this paper, it will be attempted to illustrate the possible sources of water, and the methods required to measure, control and eliminate water in quench oil to properly avoid the potential of catastrophic quench oil fires.

Methods of Water Removal

There are six primary methods for the removal of water. They include:

**Heating (Boiling off)**

Heating the oil is a very effective method to remove water, which over time will evaporate. Some oil filters can be fitted with a heating coil with the purpose to heat the oil to a temperature where water will evaporate. Free water and some dissolved water can be removed with this method.

The drawback of this method is that to be effective the oil must be heated to a high temperature, which will increase oxidation and varnish generation significantly leading to a shorter oil life and part staining. Since water is removed by evaporation the cost of power per amount of water is several times higher compared to other means of water removal.

Boiling off water is accomplished by gradually increasing the oil temperature to a little above the 100°C, with the agitators running. If in an integral quench furnace, the outer door should be opened, and the inner chamber door closed, and at heat. The inner chamber is left up to heat to prevent any moisture from contaminating the refractory. Test samples should be taken at intervals (with the agitators running) to ensure that a representative sample was taken. Initially a crackle test can be run. Once no water is detected using this method, it should be followed-up with a Karl-Fischer titration for accurate water determination.

**Coalescing**

Coalescing is a very common method of water removal from various types of oils and fuels. Water droplets are separated from the fluid in three steps.

1. The first step is the filter media which removes particles and other contaminants from the oil, increasing the interfacial tension between oil and water leading to an increase in water separation.
2. In the second step the fluid passes a fine mesh of fibers where water droplets are merged together (coalesced), here they will reach a size allowing them to be removed.
3. Separation from the fluid in the third step, where a bulk layer of water is formed in the bottom of the separator, to be discharged from the filter system.

The filter separator is only able to remove free water and to some extend emulsified water, but not dissolved water.

The efficiency of the filter separator drops with higher oil viscosity and water separation strongly depends on the interfacial tension between the oil and water. A water separation test (ASTM D 1401) of the specified oil is required to determine whether the filter separator can remove water from the oil or not.

This method of water removal is very effective, as long as the oil demulsifies. Most quench oils are paraffinomitic types, which show good demulsification. The ASTM test looks for complete separation of an oil and water mixture (40ml each) in 15 minutes, to be considered a good rating. It is important that when considering this type system, you do this test on an actual oil sample, as oil can lose the ability to shed water over time. For example, a new oil may very well pass the Demulsibility test, but because of age or surfactant contamination, the oil would trap or hold on to any water, making it a poor candidate for Coalescing. Also, some additive packages may tend to increase emulsification of the quench oil.

A typical coalescing filter is shown in Figure 5.

**Absorptive Filtration**

Filters containing cellulose, water adsorbing zeolites or other hydrophilic materials, can to some extent absorb free, emulsified and dissolved water from oil. The volume of absorbed water depends strongly on the amount of hydropilic material, the oil temperature and the water content in the oil. After some time, the filter will reach maximum capacity of absorbed water, and must be replaced.

This method is suitable for all oil types, and will also remove particles and varnish from the oil, but is most economical for
smaller systems containing little amount of water in the oil e.g. condensation accumulated over time.

This method is very effective in the day to day cleaning of your quench oils and can be looked at as a maintenance tool, more than a recovery type tool (for waste oils coming from a skimmer on the back end of a water wash application). A typical absorber type filtration system is shown in Figure 6.

**Vacuum Distillation**

When oil is put under vacuum the moisture will evaporate off at normal operation temperature. This principle is used in vacuum purifiers which can combine filtration of the oil as well. It will remove dissolved water and some emulsions, but does not perform well with free water. Foaming of the oil could occur, and result in an equipment fault.

This method is suitable for most oil types, but is most economical for complete closed systems without much water ingress. High capital cost, difficult settings and operation are some of the drawbacks. Free water can be removed with some vacuum dehydration units, but the cost of power per. removed ml. of water is several times higher compared to a filter separator or a centrifugal separator, since the vacuum dehydration unit must evaporate the water before it can be removed. This system also works best when the oil is heated to increase the vapor pressure of the water. Often, an absorption type filter is used prior to vacuum distillation.

Often times, when high levels of water are detected, the use of an outside company to vacuum distill the water is called to process the quench oil. They can process the contaminated oil quickly, as well as filter the oil, but the cost is high – typically half or more of the cost of new quench oil per gallon.

**Figure 5. Typical coalescing filter also useful for removing very fine particulate.**

**Figure 6. Typical absorber type of filtration useful for removing fine particulate and small quantities of water.**

**Centrifugal Separators**

The water is separated from the oil by centrifugal force utilizing the difference in specific gravity between the fluid and the water. Large amounts of free water and some of the emulsions can be removed per hour, but not dissolved water. Excellent water separation is required (see reference to the demulsify test above). The disadvantage is high capital and maintenance costs, as well as high power consumption under continuous operation. The heavy dirt load for a quench oil application would make this choice problematic from a maintenance perspective.

**Desorption**

Free water, emulsions and some dissolved water can be removed with desorption type systems. This system works by heating the oil and circulating dry air/gas through the oil. The dry air absorbs moisture from the oil, thereby removing water. This system is based on reliable technology which requires little maintenance, and is most economical for large systems or oil types which dissolve large amounts water and create strong emulsions.

The degree to which the oil can be dried depends on the ability of the oil to release water. A water activity test of the quench oil is required to determine whether a desorber can remove water from the oil or not.
High capital cost and high-power consumption is required to remove the water due to the necessary heating and cooling of oil and air. This method is well suited for recovering quench oil from washer solutions, or other badly contaminated solutions. With careful monitoring, the oil can be recovered for further use in a short period of time. This can represent a significant cost savings to the heat treater in further purchases of quench oil, or cost avoidance in not having to dispose of the used quench oil. If a large system is processed, the oil should be tested after recovery to verify that the water has been removed, and that the oil is suitable for continued use.

The effectiveness of a desorber in removing water is shown in Table 1. In this example, several thousand liters were processed, with the water reduced to a safe level (below 1000 ppm) in less than 9 days. An example of a Desorber Filter is shown in Figure 7.

Table 1. Results of water contamination in a quench oil, in recovered washer solution, and resulting reduction of water present using a Desorber filter. Water content determined per ASTM D6304.

<table>
<thead>
<tr>
<th>Time (days)</th>
<th>Initial</th>
<th>5</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water %wt</td>
<td>4.684</td>
<td>.8215</td>
<td>.0381</td>
<td>.0199</td>
<td>.0228</td>
<td>.0131</td>
</tr>
</tbody>
</table>

Figure 7. Typical desorber used for the recovery of water from quench oils.

A recent case study at a supplier to the automotive industry in the Midwest, resulted in water removal from almost 5% to an acceptable level of 131 ppm in their oil (Table 1). The oil performance characteristics were still intact. The financial impact for recovering this waste oil stream was a $192,000 cost savings each year. The payback time period for the equipment to remove water from this waste oil stream was less than two months.

A summary of the different methods of oil removal are shown in Table 2.

Saving waste oil

The oil that is dragged out with the parts and then removed in the wash process can be reclaimed. If the oil is intended to be reclaimed and returned to the quench tank, then the washer should be used for washing only heat treated parts and only those heat treated parts quenched in the recovered quench oil. In general, it is recommended that the oil be segregated, and processed off-line. Fine filtration of the oil is always recommended.

The oil should be tested prior to entering the quench system. This verifies that the water has been removed, and verifies that any additives have not been removed from the quench oil. Recommendations by the quench oil supplier regarding appropriate additives should be followed. Further, it is recommended that ratio of new to reclaimed oil should not exceed 50%. The primary reason is making sure that any accumulated soaps in the reclaimed oil do not cause any deleterious effects on the cooling curve. Use of new oil replenishes the additive package (speed improvers and antioxidants).

When looking at recovering a waste oil stream, the return on investment on these type systems is normally very short due to the cost savings associated with the alternative, which would be disposal costs and purchase of new oil. The oil can be separated from the wash water using a separator such as a skimmer. This oil is a waste oil because the high water content. Though using an appropriate method of water removal, the water level can be reduced to under 200 ppm, making it safe to reuse the oil.

Many companies are spending anywhere from $2.50 to $5.50 per gallon to have oil treated for reuse on-site by an outside company. Equipment leases of $1,500 to $2,500 per month to have rental equipment on site to do the treating are not uncommon. Much of these costs can be recovered by performing water recovery onsite themselves, and minimizing disposal costs.

Conclusions

Water in quench oil is a problem that often encountered and must be dealt with appropriately to reduce soft spots, distortion or even fires and explosions. Whether it is present in small amounts that may impact the life of the oil, quench curve, or part cleanliness, or in larger amounts that could lead to potential safety and health hazards or large environmental impacts, there are methods and systems available to minimize all those hazards, issues, and financial strains. Quench tanks, including storage tanks, should be evaluated to determine the current level of contamination and risk factors.

The evaluation process should consider the risks of water entrainment, and the risk of fire, explosion and injury. The evaluation should include different methods of water mitigation and examine the cost-effectiveness of each possible solution. Finally, the waste stream, and the costs associated with disposal or recovery should be evaluated. The benefits of re-use of
quench oil and the cost of any necessary additives to bring the oil back to a new condition should also be considered.

Table 2. Comparison of filtration types to remove both free and dissolved water [4].

<table>
<thead>
<tr>
<th>Filtration Type</th>
<th>Coalescing Filtration</th>
<th>Desorber Filtration</th>
<th>Absorbent Filtration</th>
<th>Centrifugal Separators</th>
<th>Vacuum Dehydration</th>
<th>Heating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Removes free water</td>
<td>Yes</td>
<td>Yes</td>
<td>Some</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td>Removes emulsified water</td>
<td>Some</td>
<td>Yes</td>
<td>Some</td>
<td>Some</td>
<td>Some</td>
<td>Yes</td>
</tr>
<tr>
<td>Removes dissolved water</td>
<td>No¹</td>
<td>Some</td>
<td>Some</td>
<td>No</td>
<td>Yes</td>
<td>Some</td>
</tr>
<tr>
<td>Min. Demulsibility requirement</td>
<td>Good</td>
<td>Medium</td>
<td>None</td>
<td>Good</td>
<td>None</td>
<td>None</td>
</tr>
<tr>
<td>Min. interfacial tension</td>
<td>&gt; 20</td>
<td>N/A</td>
<td>None</td>
<td>&gt; 25</td>
<td>N/A</td>
<td>N/A</td>
</tr>
<tr>
<td>Particle and varnish removal</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>Some particles</td>
<td>Some particles</td>
<td>Some</td>
</tr>
<tr>
<td>Power consumption vs. water removed</td>
<td>Low</td>
<td>Medium</td>
<td>Low</td>
<td>High</td>
<td>Medium</td>
<td>High</td>
</tr>
<tr>
<td>Capital cost</td>
<td>Medium</td>
<td>Medium</td>
<td>Low</td>
<td>High</td>
<td>High</td>
<td>Low</td>
</tr>
<tr>
<td>Maintenance costs (spare parts, and service required)</td>
<td>Low</td>
<td>Low</td>
<td>Low</td>
<td>High</td>
<td>Medium</td>
<td>Low</td>
</tr>
</tbody>
</table>

References


