

RECOVERY OF POTENTIALLY TOXIC COMPOUNDS FROM PHOSPHORIC ESTER-BASED HYDRAULIC FLUIDS (HFDR) USED IN THE MINING INDUSTRY

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Abstract: *The main trends are greater use of piston pumps and a sustained drive for greater power and efficiency. Piston pumps handle high flow rates at high hydraulic system pressures, thus providing optimal efficiency and reliability, while maintaining a compact size with high power density. For hydraulic systems, the main consequences of these trends have been increased operating pressures and temperatures for fluids. In addition to higher pressures and temperatures, fluids are expected to have a longer service life and be more resistant to oxidative and thermal degradation. Frequent recirculation can affect fluid performance, place additional stress on fluids, and shorten their lifespan. Formulations that provided acceptable fluid life and provided effective air release and foam control in the past may no longer achieve the same levels of performance. This is causing fluid developers to respond by developing new formulations that offer better performance under high temperature and pressure conditions. The challenge of increasing performance levels with lower fluid volumes and more rigorous operating conditions has been further complicated as some potential hydraulic fluid components have become unavailable or limited in the use level by changes in legislation. Original equipment manufacturers and end customers are very focused on fluid composition and are sensitive to potential non-fire hazards associated with fluid components.*

Keywords: HFDR, fire-resistant, hydraulic fluids, phosphoric acid

1. Introduction

In the mining sector, improving equipment reliability and operational effectiveness means reduced costs for mining. Thus, high-performance lubricants and their optimization can extend the life of lubricated components and therefore the availability of the equipment used. Reduced energy and lubricant consumption also have a positive effect on operating costs. Choosing the right hydraulic oils and corrosion-inhibiting lubricants for wire ropes is an essential but not sufficient step. Improving lubricants developed specifically for this type of industry makes mining safer, more efficient and less expensive. Therefore, the composition of these hydraulic oils is special, which takes into account the harsh working conditions and their potential contaminants that can damage the equipment. Whether it is abrasive contamination, external temperatures or oscillating movements and extremely high loads, the products used in this field have high load capacity and protection against wear, wet or dry conditions, corrosion, or the working temperature regime.

Manufacturers' recommendations for optimal product selection indicate special caution in taking into account operating conditions but also other parameters such as working time, the nature of the lubricants, their composition and especially the process and equipment for which they are used.

The latest generation oils, special and specific to this industrial sector, mining, represent a constant challenge for both manufacturers and operators, to ensure the efficiency and reliability of the equipment necessary for the work in the field. In addition to concerns regarding sustainability strategies for energy consumption, CO₂ emissions, product life cycle costs, compliance with production standards and requirements, reliability and noise reduction, resistance against external or internal influencing factors, the circularity of used lubricating and industrial oil management has also become an equally important aspect. Therefore, the specifications issued by professional manufacturing companies, hydraulic equipment manufacturers and standardization organizations provide indications on the chemical composition and physical-chemical properties of the type of hydraulic fluid, the applications, specifications and approvals in force, the benefits but also instructions on its management. These represent a way of selection and adaptability to the user's needs.


2. Materials and methods

For mining, the exploitation activity takes place in a wide thermal range (-20_150 °C), as such, non-flammable oils are chosen, classified ISO-L-HEES, ISO-L-HFC and ISO-L-HFDU (antioxidant / antirust / antiwear), characterized by reduced viscosity variations in relation to temperature, which ensure adequate lubrication under conditions of high thermo-mechanical stress.

Biodegradable Fluid

ISO-L-HEES e ISO-L-HFDU

Caratteristiche fisiche /	Metodo /	Valore /
Physical Characteristics	Method	Value
Densità / Density (@15°C)	ASTM D405	0,925 kg/dm ³
Viscosità / Viscosity (@40°C)	ASTM D445	46 cSt
Viscosità / Viscosity (@100°C)	ASTM D445	9,5 cSt
Indice di viscosità / Viscosity index	ASTM D2270	186
Infiammabilità P.M. / Flash point	ASTM D92	>300°C
Punto di scorrimento / Pour point	ASTM D97	-36°C
Acidità / Acidity	ASTM D 974	2,2 mg KOH/g
FZG Stadio / FZG Test-Studio	ISO 14635	>=12
Rilascio aria / Air release	ISO 9129	<1 min
Biodegradabilità / Biodegradability	OECD 301B	>70%




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Fig. 1. Technical Data Biodegradable Hydraulic Fluid_GWV

PHYSICAL CHARACTERISTICS	METHOD	VALUE
Density	ASTM D405	0.925 Kg/dm ³
Viscosity at 40° C	ASTM D445	40 cSt
Viscosity at 100° C	ASTM D445	9,5 cSt
Viscosity index	ASTM D2270	186
Flash point	ASTM D902	>300° C
Pour point	ASTM D97	-36°C
Acidity	ASTM D974	2.2mg KOH/g
FGZ Test-Studio	ISO 14635	≥ 12
Air release	ISO 9129	<1 min
Biodegradability	OECD 3018	>70%

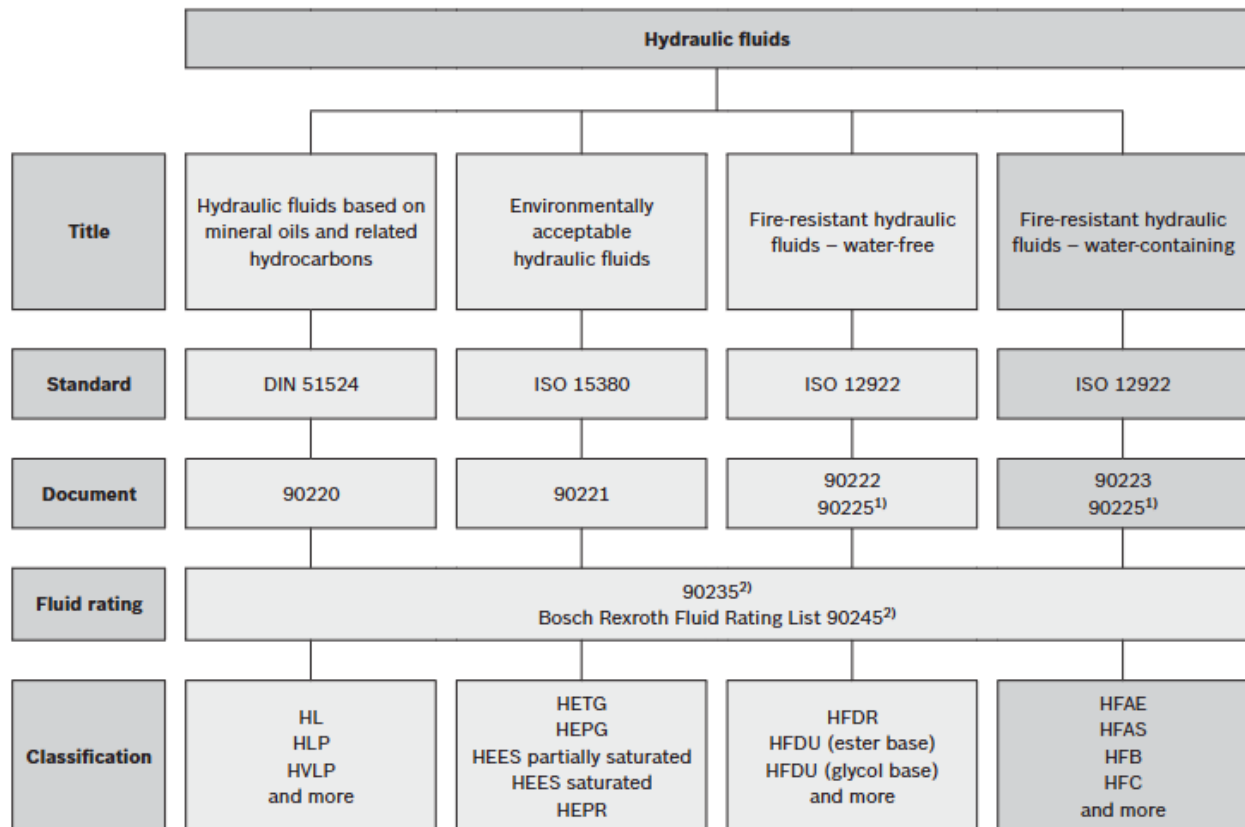
Fig. 2. The physical characteristics of Biodegradable Hydraulic Fluid_GWV

Fluids with high resistance to ignition and difficult flame propagation are compounds based on phosphoric esters (HFDR), characterized by a density that slows down the response speed of the hydraulic system.

This type of high-performance hydraulic oils are designed to meet the stringent requirements of modern models from original equipment manufacturers. It must also provide protection for mobile and stationary hydraulic pumps with vanes, pistons and gears, servo valves using multi-metallic components, operating in industrial, mining and marine applications, in environmentally sensitive areas, where the impact of hydraulic oil on the environment is a real concern.

The characteristics of these hydraulic oils consist of advanced hydrolytic stability which helps prevent corrosive wear, effective oxidation stability which provides optimal performance throughout the entire operating period, they are formulated to control deposits which helps maintain system accuracy and reliability. These characteristics lead to good productivity, long system life but also low toxicity for the water fractions tested. The main role of hydraulic fluid is to transmit power, lubricate components, reduce friction, prevent corrosion, and dissipate heat.

The demanding requirements regarding machinery and equipment constantly require the fulfillment of specific requests regarding the quality of the hydraulic fluid used, requiring knowledge and experience to identify and properly select a hydraulic fluid.



1) Valid for Bosch Rexroth axial piston units

2) Valid for Bosch Rexroth Business Unit "Mobile Applications" – pumps and motors

Fig. 3. Classification of hydraulic fluids - RE 90222 Fire-resistant, water-free hydraulic fluids (HFDR/HFDU); Source: Bosch Rexroth AG, RE 90223/01.2015

Water-free, fire-resistant hydraulic fluids hydraulic components are assessed on the basis of their fulfillment of the minimum requirements of ISO 12922. Hydraulic fluid suitability depends, amongst others and the viscosity factor, it being a basic property of hydraulic fluids.

The permissible viscosity range of complete systems needs to be determined taking account of the permissible viscosity of all components and it is to be observed for each individual component. The viscosity at operating temperature determines the response characteristics of closed control loops, stability and damping of systems, the efficiency factor and the degree of wear. The optimum operating viscosity range of each component be kept within the permissible temperature range. This usually requires either cooling or heating, or both. If the viscosity of a hydraulic fluid used is above the permitted operating viscosity, this will result in increased hydraulic-mechanical losses. In return, there will be lower internal leakage losses. If the pressure level is lower, lubrication gaps may not be filled up, which can lead to increased wear. For hydraulic pumps, the permitted suction pressure may not be reached, which may lead to cavitation damage. If the viscosity of a hydraulic fluid is below the permitted operating viscosity, increased leakage, wear, susceptibility to contamination and a shorter component life cycle will result. It is required that the permissible temperature and viscosity limits are observed for the respective components.

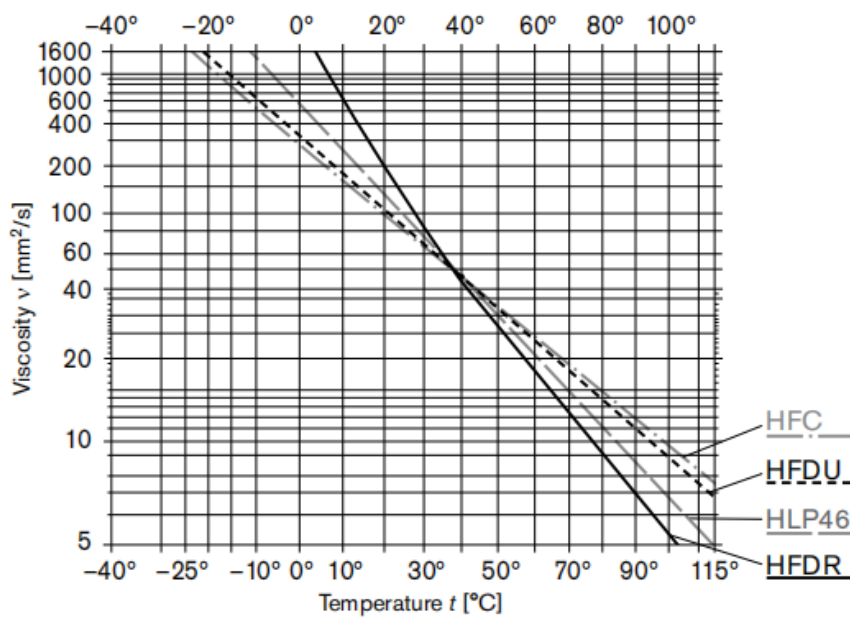


Fig. 4. Diagrams for water-free, fire-resistant hydraulic fluids in comparison to HLP and HFC (reference values, double-logarithmic representation)

Table 1: Hydraulic fluids, the viscosity temperature behaviour (V-T)

Typical viscosity data [mm ² /s]			
at temperature	0 °C	40 °C	100 °C
HFDR	2500	43	5,3
HFDU (ester base)	330	46	9,2
For comparison HLP (see RE 90220)	610	46	7
For comparison HFC (see RE 90223)	280	46	

For hydraulic fluids, the viscosity temperature behaviour (V-T) is of particular importance. The interrelation between viscosity and temperature is described by the viscosity index (VI).

For cold testing over a period of several days, the viscosity of ester-based HFDU can increase greatly. HFDU fluid based on ester have better viscosity/temperature characteristics than mineral oil HLP (Fig. 4).

Ageing resistance is the way a water-free, fire-resistant hydraulic fluid ages depends on the thermal, chemical and mechanical stress to which it is subjected. The influence of water, air, temperature and contamination may be significantly greater than for mineral oils HLP/HVLP. High fluid temperatures (e.g. over 80 °C) result in an approximate halving of the fluid service life for every 10 °C temperature increase and should therefore be avoided. Ageing resistance can be greatly influenced and by the chemical composition of the hydraulic fluids.

3. Results

Phenomena within the lubricant layer have a great influence on lubrication, which increases with the thickness of the lubricant layer. These phenomena involve associations of molecules under the action of existing polar substances, but also through internal friction between molecules.

Thus, viscosity is very important in determining the lubricating capacity of lubricants.

Considering the chemical composition of lubricants based on phosphoric acid esters such as tricresyl phosphate, trioctyl phosphate, or diethyl ester of decanephosphonic acid, as well as polymeric tetrahydrofurans, it is necessary to process used oils, thus facilitating their regeneration and recovery process. By removing impurities and other unwanted substances, treated oils with improved quality are obtained, ready for re-introduction into the industrial circuit or for use as raw material in the petroleum industry. Thus, a wide range of aqueous liquid waste resulting from various industrial activities can be processed. The state-of-the-art facilities used are designed to efficiently manage liquid waste, ensuring the neutralization of toxic substances and minimizing the impact on the environment. The processing of used oils and aqueous waste must comply with the highest environmental and safety standards, to ensure their compliance with regulations in force.

The companies' commitment to innovation, sustainability and efficiency is reflected in every aspect of the services offered, with the companies becoming trusted partners for those who aim to manage waste responsibly and sustainably. In the specialized literature, methods and processes are described regarding concerns for recovering phosphoric acid from waste oils, being used due to its liquid state and easy injection into various equipment used in fields such as mining, agriculture, irrigation, etc. Phosphoric acid can be obtained from phosphates, commonly from apatite $\text{Ca}_5(\text{PO}_4)_3(\text{F}, \text{OH}, \text{Cl})$ by reaction with stronger acids (sulfuric acid or nitric acid).

The anhydrous form of phosphoric acid is hygroscopic, and the hydrolysis reaction yields phosphoric acid, the by-products that arise are CaSO_4 and hexafluorosilicic acid H_2SiF_6 .

These involve treating phosphoric acid with a mixture of at least two extraction solvents at a temperature low enough to form a clear, homogeneous extract. By controlling the temperature, a separation of the phases can be made, namely, the residual aqueous phase and the extract phase into a lower layer containing phosphoric acid and an upper layer containing solvents and separating the lower layer from the upper layer. Solvents are, for example, esters, ketones, glycol ethers and ethers, such as ethyl, butyl and amyl acetate, ethyl butyrate and cyclohexanone.

Stratification is improved by adding a small amount of purified phosphoric acid. The yield is increased by incorporating 1-5% sulfuric acid into the technical grade acid. The residual acid, after separation of the solvent extract, is acidified with sulfuric acid in an amount of 30 to 100% by weight of phosphoric acid and subjected to repeated extraction with a solvent or with a mixture of extraction solvents and a mixture of purified phosphoric and sulfuric acids.

This recovery method, however, contains some phosphoric acid impurities that are difficult to remove. One of the removal processes consists of adding alumina, calcium compounds, activated carbon dioxide, silica, or activated clay before solvent extraction. Calcium hydroxide or some phosphate-type compounds such as phosphate rock can be added to phosphoric acid to neutralize the free sulfuric acid present after treatment for better separation of the extraction phase.

The solvents used are: ethyl ether and n-butyl ether; n-butyl ether and dibutyl ether of ethylene glycol, isopropyl ether and ethyl ether, isopropyl ether and cyclohexanone, ethyl ether and

monohexyl ether of diethylene glycol, mixed mono- and di-n-butyl ethers of diethylene glycol, diethylene glycol dibutyl ether and diisobutyl ketone.

Therefore, a residual fraction from the extraction of phosphoric acid is treated in the first phase with a mixture of n-butyl ether and ethyl ether, then, the extraction phase is acidified with concentrated sulfuric acid and the mixture is extracted with a mixture of isopropyl ether and ethyl ether.

Therefore, the purification of phosphoric acid by extraction can be used in the chemical industry.

Another organic solvent-based phosphoric acid extraction process consists of adding a base of monovalent cations, such as sodium, potassium, ammonium and their derivatives or salts thereof, with phosphoric acid in a molar ratio of the monovalent cation added to the sulfate ion in the phosphoric acid of 0.5-10%. The concentration of phosphoric acid in the aqueous phase is 1.5-10.5 mol/g. The extract is washed with an aqueous solution of the same substances.

The organic solvents used are preferably ketones and ethers, for example, tributyl phosphate in pure form with the addition of a polar diluent. The method makes it possible to perform a more efficient purification of phosphoric acid by removing sulfuric acid from the separation phase. This process, like many other methods, does not have good environmental compatibility.

As such, concerns in this direction have led to another method of recovering phosphoric acid, an environmentally friendly method. This method involves the recovery of phosphoric acid in a humid environment, being a method of purifying phosphoric acid through solvent extraction.

The method targets one or more systems of ketone, ether and ester extraction agents, and the purification process mainly comprises the following steps: sequential extraction, washing and reverse extraction of crude phosphoric acid. The process consists of a pre-emulsification stage of ether solvents and carboxylates, then mixing with mineral oil and its inorganic salt and finally emulsification, homogenization and thinning to obtain the antifoaming agent - the wet medium for phosphoric acid extraction. In the purification process, the residual washing acid and the reverse extraction acid are concentrated before being recycled, so that the residual washing acid and the reverse extraction acid are controlled in relation to the phosphoric acid concentration in the corresponding extraction system. The generated residual wash acid is returned to be mixed with wet process phosphoric acid and concentrated to serve as crude phosphoric acid, and the reverse extraction acid thus generated is partially concentrated and returned to serve as wash acid.

The method is used for extraction systems of ether, ketone and ester extraction agents.

The extraction rate of phosphoric acid can be remarkably increased, the loss of phosphoric acid and the amount of acid phase circulation in the washing process are reduced, and the yield of phosphoric acid is high. The process is simple, easily controllable due to the mild conditions for separation and extraction of phosphoric acid. Production costs and energy consumption are reduced. The process does not pollute the environment, and the product obtained has a low surface tension, with a foam inhibition effect, the performance of obtaining the product is high.

The use of wet media has proven to be suitable not only for the production of phosphoric acid but also for the elimination of acidic medium foam and the inhibition of foam in the spinning, printing and dyeing industries and the like. In the wet process variant, elements such as iron, aluminum and magnesium can also be recovered. These can be expressed as oxides, from phosphoric acid with minimal phosphate losses and dilution to produce a phosphoric acid that is suitable for the production of fertilizer products such as diammonium phosphate (DAP), commercial grade phosphoric acid, superphosphoric acid and other phosphoric acid products using a continuous ion exchange approach. Furthermore, the method allows the use of lower grade phosphate rock or ore in the extraction process, which would considerably expand the potential phosphate rock reserve base for phosphate mining activities and allow for better overall utilization of resources from a given developed mining site. Phosphoric acid, produced from alumina-rich phosphate rocks, is a by-product of phosphate rock mining operations. The rock is finely ground and dispersed in sulfuric acid, and the resulting phosphoric acid contains the metal ions normally present in the treated rock.

The metal ions are then extracted from the acid by ion exchange with a water-immiscible organic sulfonic acid compound, preferably in the presence of an organophosphate or phosphonate.

After phase separation, the organic phase containing the extracted metal ions can be regenerated.

The process is particularly useful when digestion is done at a P_2O_5 concentration and temperature that produces calcium sulfate hemihydrate. By optimizing the process, production efficiency is greatly improved. The wet phosphoric acid production method is a new method of producing phosphoric acid by purifying with wet process phosphoric acid. The P_2O_5 content of wet process phosphoric acid is higher, and phosphoric acid contains a lot of SO_4^{2-} ions.

The P_2O_5 content of the acidosis liquid produced by the decomposition of phosphate ore by sulfuric acid is high. The wet process phosphoric acid and the acidolysis liquid are mixed, so that the P_2O_5 content in the mixed liquid is improved, and the content of calcium ions and sulfate radicals in the mixed liquid is reduced. The new method has a certain promoting effect on the separation of other impurities. According to the new method, mixed desulfurization is used to replace the conventional desulfurization technology with phosphoric ore paste and barium carbonate, so that the single-stage desulfurization rate can reach over 99%. Two different phosphoric acids are mixed so that SO_4^{2-} and Ca^{2+} generate precipitates of $CaSO_4$ to eliminate SO_4^{2-} .

The optimal decontamination index (W) of the obtained mixed acidic liquid is $w(H_3PO_4)=10-25\%$, while $w(SO_4^{2-})$ requires continuous monitoring. The main component of the filter residue is $CaSO_4$, and the purity is high and reaches 99%. The mixed extract for the purification of wet phosphoric acid is a mixture of n-butanol (60-70 vol%), tributyl phosphate (20-30) and methylisopropyl methanone (10-20) with high extraction and impurity removal power.

Purified phosphoric acid can be used to prepare nano-grade silver chloride by adding silver nitrate. Purified phosphoric acid contains fluorine produced from wet process phosphoric acid which can be divided into two parts, one part is used as returned acid and the other part is used as crude phosphoric acid. This technology involves a device for producing defluorinated phosphoric acid.

The device includes 2 injection pumps, a reaction kettle, a gas-liquid separator and an absorption tower where they are sequentially connected by pipes to form a closed, circulating pipe system that is in a negative pressure state. The 2 injection pumps allow the gas in the system to flow circularly at high speed. The injection pump sucks and mixes the returned acid and sulfuric acid, then sucks a reaction liquid and sulfuric acid into the reaction vessel, where after mixing them with the gas flow, the resulting mixture is sprayed onto the liquid level of the reaction vessel. The vacuum generated by the other injection pump causes the crude phosphoric acid and the low-fluorine hot steam emitted from the reaction vessel to be sucked in and mixed.

The resulting mixture enters the gas-liquid separator, then the gas is sent to the bottom of the absorption tower, and the defluorination waste gas is led from the top of the absorption tower to the injection pump. The washing liquid is sprayed from the top of the absorption tower and the fluosilicic acid liquid is led out through the bottom of the absorption tower. This liquid flows circularly after exiting the separator, being a refined defluorinated product of phosphoric acid.

Fluorine in the form of fluoride is removed from phosphoric acid having an initial phosphate concentration of less than about 47% by determining the concentration of fluorine to be removed from the acid and the concentration of silicon in the acid. Silicon is added to the acid in an amount sufficient for the molar ratio of fluorine to be removed from the acid to silicon to the acid. The acid is concentrated so that the fluoride reacts to fluosilicic acid. By maintaining the indicated concentration ratio of fluorine to be removed and silicon, fouling of the condenser and scrubber components with deposited silica is avoided. Silicon-containing material is added to the phosphoric acid preparation process from phosphoric rocks so that the aluminum content in the wet phosphoric acid process is reduced. When the temperature of the phosphoric acid reaches the reaction temperature of 78-85°C, powdered rock phosphate and silicon-containing material are added to the phosphoric acid simultaneously. Under continuous stirring, the reaction takes place for 5-7 minutes, then concentrated sulfuric acid with a mass fraction of 98% is added and stirred to react sufficiently, and the reaction time is 1-3 hours, then filtration is performed and wet process phosphoric acid is obtained. The method is simple and easy to use, efficient and feasible for the reduction of aluminum ions in the wet phosphoric acid process. Since the discovery of their excellent anti-wear and fire resistance properties, the use of phosphate esters by industry has steadily increased (Figure 5).



Fig. 5. Benefits and limitations of phosphate ester fluids used in industry

Source- <https://www.machinerylubrication.com/Read/2480/benefits-limitations-of-phosphate-ester-fluids>

Phosphate esters are primarily used as fire-resistant base materials in several applications, especially in hydraulic systems, turbines and compressors. Phosphate esters are the most fire-resistant of the non-aqueous synthetic base materials. Numerous organic phosphorus compounds, including phosphites, phosphonates, and phosphates, have found applications as additives in a variety of lubricant formulations as stabilizers, anti-wear additives, antioxidants, metal passivated, and extreme pressure additives. Only one group of phosphates, the natural trisubstituted esters of H_3PO_4 , has found significant use as a synthetic base. The use of phosphoric ester-based products in hydraulic applications is still primarily dictated by fire risk considerations. Although inhibited phosphate esters possess excellent oxidation stability and inherently good anti-wear properties under critical loading conditions, they suffer from somewhat inferior hydrolytic stability.

As such, there are concerns regarding this aspect and others such as optimizing the viscosity index and reducing chemical aggressiveness compared to some conventional sealing and coating materials. These points still limit the use of phosphate ester to specialized applications where a high degree of fire resistance is required.

4. Conclusions

The paper aims to describe methods for chemical stabilization and reprocessing of residual materials from hydraulic oils used in mechanical management processes (recovery, transport and processing) of the tailings of an ore deposit using efficient technologies in order to reduce the concentration and toxicity of their composite elements. The possibility of recovering and treating phosphoric acid from hydraulic oils for use as a raw material in other processes represents a way to capitalize on them. The method described in this paper represents a new method for producing phosphoric acid by purification with wet process phosphoric acid.

The method makes it possible to perform a more efficient purification of phosphoric acid by removing sulfuric acid from the separation phase. The new method has a certain promoting effect on the separation of other impurities as well. According to the new method, the mixed desulfurization rate in a single stage can reach over 99%. The method is simple and easy to use, effective and feasible for reducing potentially toxic elements in the wet phosphoric acid process.

Acknowledgments

The work was carried out through the Core Program within the National Research Development and Innovation Plan 2022-2027, carried out with the support of the Romanian Ministry of Research, Innovation and Digitalization (MCID), project no. PN 23 05.

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