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EXPERIMENTAL COMPARISON OF VEGETABLE AND PETROLEUM BASE OILS IN METALWORKING FLUIDS USING THE TAPPING TORQUE TEST

Andres F. Clarens¹ Julie B. Zimmerman² Heather R. Landis³ Kim F. Hayes¹ Steven J. Skerlos³

1. Department of Civil and Environmental Engineering
3. Department of Mechanical Engineering
The University of Michigan at Ann Arbor

2. Office of Research and Development
Environmental Protection Agency

ABSTRACT

Traditional metalworking fluid (MWF) formulations have been associated with a number of environmental and health concerns that have driven recent efforts to develop new formulations based on alternative vegetable and ester based feed stocks. This study uses the tapping torque test to compare the performance of five base oil feedstocks for MWFs: naphthenic mineral oil, a 50/50 blend of naphthenic and paraffinic mineral oil, soybean oil, canola oil (75% oleic content), and a TMP Ester. The five oils were tested as straight oils, and as soluble oil and semi-synthetic MWFs, to understand the impacts of emulsification on base oil performance. Machining performance was evaluated using a modification to the standard tapping torque test (ASTM D 5619) previously established by the authors. Over 500 tapping torque experiments are represented in this research. The results indicate that as straight oils all vegetable based stocks perform significantly better than the mineral oils. This trend holds, although is much less pronounced, after the vegetable stocks are emulsified into soluble oil and semi-synthetic MWFs. The results also indicate that some vegetable oil base stocks have a higher potential for lubricity than others, with data revealing that the soy and TMP ester provide improved tapping torque efficiency relative to canola oil in emulsified MWFs.

KEY WORDS

Metalworking fluids, vegetable base oils, EP additives, particle size, tapping torque test

INTRODUCTION

Sustainable aqueous systems minimize life cycle environmental impact by 1) minimizing the materials, energy, and toxicity of system inputs and outputs, and 2) achieving maximum system lifespan by maintaining physical, biological, and chemical parameters within limits appropriate to system

function. Although it is estimated that over 1 billion gallons of *metalworking fluids* (MWFs) are consumed each year, metalworking fluid systems are universally in violation of these principles, and the need to develop basic knowledge and technology to achieve sustainability has been increasingly recognized in recent years.

By serving as both coolants and lubricants, metalworking fluids (MWFs) are critical to a wide range of manufacturing operations [1]. However, MWFs are harmful to the environment due to their high oil content, biochemical oxygen demand, surfactants, and because they serve as carriers for hazardous metals and chemicals [2]. Moreover, the U.S. EPA has proposed regulations that would limit the discharge of oil and grease to 35 mg/l [3]. This is significant because MWFs can contain over 6,000 mg/l of these constituents, and meeting the standard would require large investments in end-of-pipe treatment technologies. Despite these environmental and health risks, and high disposal costs, MWF use remains strong and continues to grow [4].

Mitigation of these financial, environmental, health, and performance liabilities requires innovative eco-design of MWF formulations toward the development of *sustainable metalworking fluid systems* (Figure 1). This includes a re-evaluation of the chemical constituents found in MWFs. The principal components of the most common MWFs used today (i.e., soluble oils and semi-synthetics) are petroleum-based mineral oil for lubrication emulsified in water for cooling. Recently, there has been increasing interest in developing "green" MWFs derived from renewable bio-based oils and more environmentally benign additives [5]. Numerous bio-based oils are available on the market, and the authors have shown in [6] for the case of canola oil-based MWFs that such vegetable oil feedstocks are likely to represent an environmentally preferable alternative to mineral oil. This is true particularly when greenhouse gas emissions are considered

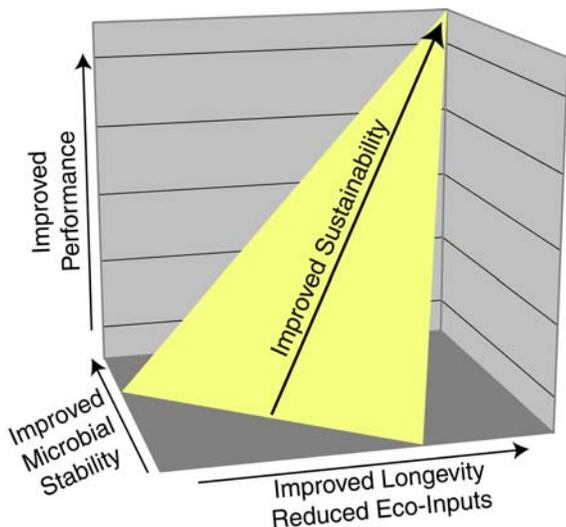


Figure 1. Target objectives for sustainable MWF systems

because carbon dioxide is sequestered when the vegetable feedstocks are grown. Problematic characteristics of MWFs, such as odors and degradation caused by biological growth, cannot be predicted nor evaluated prior to formulation, although preliminary studies suggest that bio-based fluids do not possess inherent disadvantages in these areas relative to petroleum based MWFs [5]. For instance, the vegetable based emulsifier systems currently being considered for MWFs tend to be more hard water stable, and biological stability has been shown to correlate closely with particle size [10], dispelling the popular sentiment that bio-based fluids are inherently more susceptible to microbial attack. Moreover, vegetable based MWFs might hold other advantages in manufacturing environments. For instance, they may be less likely to produce harmful process by-products such as aromatic hydrocarbon aerosols.

While interest in bio-based oils as feedstocks for MWFs is increasing, little information is available in the literature regarding their performance as MWFs relative to conventional alternatives. Therefore this paper compares the relative performance of five base oils in straight and emulsified form for their performance in the tapping torque test. Specifically, three common bio-based oils available on the market (canola, soy, and a synthetic ester) were tested and compared to two common mineral oils used in lubricants (a naphthenic and a 50/50 naphthenic/paraffinic blend). The performance of these base stocks is evaluated in tapping operations involving both 1018 and 4140 steel workpieces. Results describing the significance of the tapping torque efficiency metric in terms of its correlation with known field performance are also provided, along with a discussion regarding the potential roles of emulsion particle size and extreme pressure (EP) additives in modifying the observed tapping torque trends for petroleum and vegetable base oils.

EXPERIMENTAL MATERIALS AND METHODS

Formulations. The MWF formulations considered here were based on a generic formula provided by a commercial MWF supplier. The MWFs were first produced in concentrated

form, and then were diluted to a working concentration in deionized water. This formulation procedure is consistent with the manner in which MWFs are prepared and utilized in practice. All MWF concentrates consisted of 1.5 wt% coupler (butyl carbitol), 3.7 wt% tall oil fatty acid, 7.9 wt% corrosion inhibitor (monoethanol amine), 15 wt% oil, and 14 wt% surfactants, and 57 wt% deionized water. For comparative purposes, the MWFs investigated differed only in base oil and surfactant system chemistry. The surfactant system chosen for each base oil is listed in Table 1 (as concentrate). Three basic MWFs were developed for each base oil, a straight oil (with no deionized water, surfactants, or other MWF additives), a soluble oil (concentrate diluted 77% in deionized water), and a semi-synthetic (concentrate diluted 95% in deionized water). As tested, the MWFs either contained 100% oil (straight oil), 3.4% oil (soluble oil), or 0.75% oil (semi-synthetic). Within a class of MWF, (straight oil, soluble oil, or semi-synthetic) the oil concentrations were always held equal to permit comparison of the base oil functionality. It was found for these MWFs that the additives listed above (other than oils and surfactants) had little impact on emulsification, performance, and stability.

Materials. For the five different base MWF formulations, all of the fluid components were used as delivered from the manufacturer and were subject to the same handling and storage conditions.

Oil type	Oil % weight in concentrate	primary surfactant type	primary surfactant % weight in concentrate	secondary surfactant type	secondary surfactant % weight in concentrate
Naphthenic	90	Tagat V 20	4.5	Alfonic 1216 CO-1.5 Ethoxylate	5.5
50/50 Blend	90	Tagat V 20	4.5	Alfonic 1216 CO-1.5 Ethoxylate	5.5
Canola Oil	83	Tagat V 20	12.75	Tegin OV	4.25
Soybean Oil	83	Tagat V 15	12.25	Tegin OV	4.75
TMP Ester	90	Tagat V 20	10	none	none

Table 1. Oils and Surfactants used to make MWF Emulsions

The base oils used in the formulations were a petroleum-based naphthenic oil, a petroleum-based 50/50 naphthenic/paraffinic oil blend, a bio-based high oleic canola oil that was modified for oxidative stability (Agri-Pure 75, Cargill Inc., Minneapolis, Minnesota), a bio-based soybean oil (Alkali Refined Soybean Oil, Cargill Inc., Minneapolis, Minnesota), and a bio-based synthetic TMP ester (Priolube 1427 Trimethylolpropane triolate, Uniqema, New Castle, Delaware). Combinations of four different surfactants were used to make stable emulsions of the oils. The surfactants were Tagat V20, Tagat V15 and Tegin OV (Degussa-Goldschmidt Chemical Corporation, Hopewell, Virginia) and Alfonic 1216 CO-1.5 Ethoxylate (Sasol North America, Austin Texas). While it would have been preferable for comparisons to use identical surfactant systems for each base oil, it was verified that it is not possible to utilize a single surfactant system for the emulsification of these different base oils. This is a deep rooted and well known conclusion often encountered in the emulsion science literature, which considers the functionality of

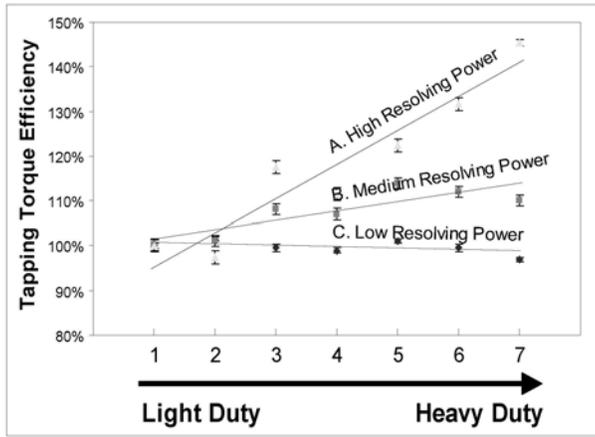


Figure 2. Non-normalized tapping torque values for 7 cutting fluids ranked from light duty to heavy duty by major supplier of MWF. (A) High resolving power condition. (B) Medium resolving power condition. (C) Low resolving power condition. After Zimmerman et al., 2003 [9]

surfactants in the stabilization of one immiscible material as a suspension in another.

The base oil formulations were made by holding the molar concentration of oil constant across all fluids. The concentrations of mineral oil in the generic recipes were used as the reference amounts of oil in the formulations. A commercially available soluble oil MWF (C225, Chrysan Industries, Plymouth, MI), was used as a benchmark to compare the oils and MWFs in the tapping experiments. C225 was utilized as it has served as a reference standard for the authors over a period of three years. The formulation for C225 was not included in Table 1 to maintain confidentiality for the manufacturer.

Methods. Emulsions were considered stable if they maintained a consistent oil-in-water particle size over a one week time period. Particle size was evaluated using photon correlation spectroscopy (PCS), which allows for the detection of subtle fluid particle size changes, including indications of coalescence. For this research, a Nicomp 370/DLS (Particle Sizing Systems, Santa Barbara, California) particle sizing system was used, with its particle size estimation capability verified independently by a wide-angle laser light scattering apparatus similar to the one described by Lee et al. [7]. Two aliquots from each formulation were analyzed by PCS and averaged.

The machining performance of the MWFs developed during this research was measured via the tapping torque test using a MicroTap Mega G8 (Rochester Hills, MI) machine tool at a machining speed of 1000 RPM on 1018 and 4140 steel workpieces that were pre-drilled and pre-reamed with 240 M6 holes (Maras Tool, Schaumburg, IL). Tapping was performed using uncoated high-speed steel taps (for 1018 steel) and CrN coated HSS taps (for 4140 steel), both with 60° pitch and 3 straight flutes. MWF evaluations were carried out according to ASTM D 5619, the Standard for Comparing Metal Removal Fluids Using the Tapping Torque Test Machine [8] with several modifications made to account for the use of a MWF evaluation testbed that permits multiple evaluations on a single workpiece as proposed by Zimmerman et al. [9]. MWF performance is reported here as percentage tapping torque efficiency (η),

which is an average torque measured during tool engagement normalized to the average torque measured for a reference MWF. Higher efficiency indicates improved performance in the tapping torque test, and has been shown to be an adequate metric for field performance as discussed below. Method details are provided in [9].

Methods: Correlation of Tapping Torque Test with Field Performance [6]. As described in [9], ASTM D 5619 was designed for tapping torque test (T^3) systems that conduct a single tapping evaluation (SES) per workpiece. As might be expected, performing each test evaluation on a new workpiece introduces significant uncertainty into the evaluation process since workpiece to workpiece variation can overshadow differences in torque responses caused by MWFs. Difficulties with workpiece variation and per-test cost have led to the development of tapping torque testbeds that allow multiple test conditions to be evaluated on a single workpiece (MES). While this makes T^3 potentially more convenient and cost effective by reducing variability associated with workpiece material, this type of system introduces new challenges in experimental design and interpretation given the potential for tool wear and localized hardness within a single workpiece.

Challenges with MES, and experimental design considerations to overcome them are described in [9]. The research indicated that depending on how the tapping torque test is performed, operating conditions can either mask MWF performance (low resolving power) or provide the means to differentiate between MWF types (high resolving power). If T^3 experimental conditions with low resolving power are selected, it may be impossible to differentiate MWFs, even where known differences exist. To illustrate this, seven MWFs varying from light duty to heavy duty were examined using MES T^3 . The MWF duty ratings were based upon the extensive field experience of a major MWF formulator working in a range of end-users. For T^3 experiments performed with M6 TiN tools at 1000 RPM (a condition found to have low resolving power), it was found that even the MWF with the lowest duty rating could not be statistically distinguished from the MWF with the highest duty rating (Figure 2C). However when conditions were chosen with a higher resolving power (e.g., M4, HSS tools, 1000 RPM), fluid differences were easily distinguishable and the T^3 responses were better correlated with the expected trend of MWF field performance (Figure 2B). At the test condition with the highest resolving power (M6, HSS, 1000 RPM), the expected field performance was captured even more clearly (Figure 2A). Based on Figure 2 different base oils are compared below via T^3 under high resolving power conditions.

RESULTS AND DISCUSSION

Results: Impact of Base Oil and Emulsification on T^3

Figure 3 presents the tapping torque efficiency for the five straight oils. The fluids were tested on 1018 cold rolled steel using an uncoated hardened steel tool. Under the tapping torque conditions of Figure 2A, the bio-based straight oils demonstrated significantly higher tapping torque efficiency relative to the petroleum oils. Both mineral oils had a slightly lower efficiency level relative to the reference (petroleum based, C225) soluble oil (efficiency < 100%). In contrast, the three bio-based oils exhibited a 12-14% increase in efficiency relative to the reference soluble oil.

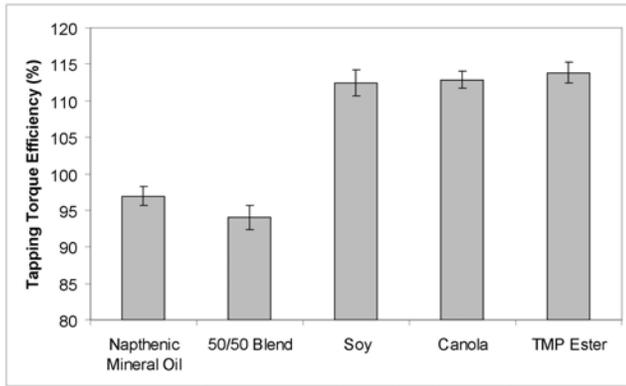


Figure 3. Tapping torque efficiency for five different base oils (napthenic mineral, napthenic/paraffinic blend, canola, soybean, and TMP ester) as straight oils (100% wt).

For the soluble oil (3.4 oil % wt) and semi-synthetic (0.75 oil % wt) formulations of these base oils, the difference in tapping torque efficiency between the petroleum and vegetable oils was found to be reduced significantly. The vegetable oils, however, still presented a significantly higher tapping torque efficiency when compared with the mineral oil based MWFs. Interestingly, the mineral oil semi-synthetic MWFs performed almost equally as well as the soluble oil MWFs in spite of the much lower oil concentration in the formulation. Figure 4 presents the tapping torque efficiency for the five oils as soluble oil and semi-synthetic emulsions. It is seen that the basic trends relative to Figure 3 are unchanged.

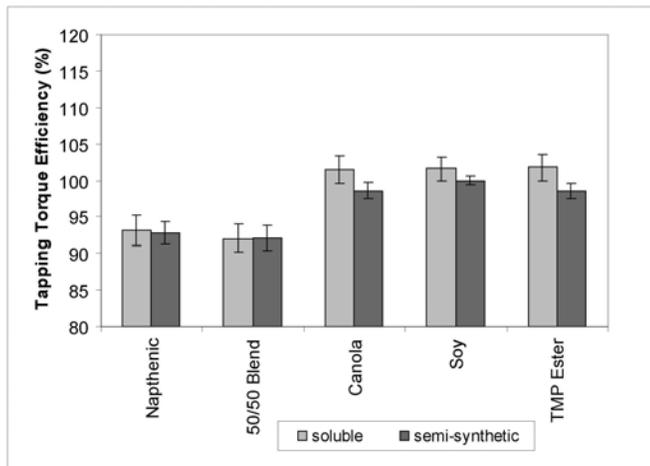


Figure 4. Tapping torque efficiency for five different base oils (napthenic mineral, napthenic/paraffinic blend, canola, soybean, and TMP ester) as soluble oil (3.4% wt), and semi-synthetic (0.75% wt) emulsions.

In order to verify these trends in the cutting of harder steels, and in the use of alternative tools (CrN coated steel), three soluble oils (based on soybean oil, mineral oil, and TMP ester) were tested via T^3 on a 4140 steel workpiece (Figure 5). It is observed in Figure 5 that similar results were obtained when tapping 4140 steel as compared with results obtained when tapping 1018 steel (Figure 4). Once again, the fluids based on bio-feedstocks exhibited a higher tapping torque performance relative to the mineral oil based fluids. However, consistent with the trends presented in Figure 2, the differences

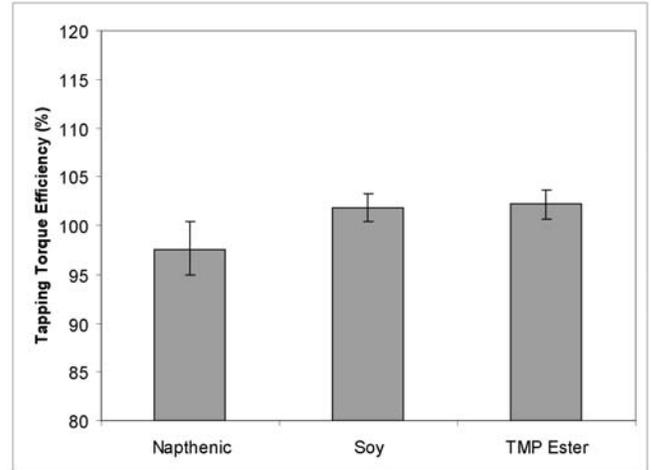


Figure 5. Tapping torque efficiency for three different base oils (napthenic mineral, soybean, and TMP ester) as soluble oil (3.4% wt) on a 4140 steel workpiece.

in tapping torque efficiency are reduced, as expected given that the resolution of coated tools has been observed to be generally less than uncoated tools [9].

Results: Impact of Secondary Additives on Lubricity

Figures 3-5 provide a comparison between the base oils (including their emulsifying nonionic surfactant and anionic surfactant) as listed in Table 1 with respect to their tapping torque efficiency. In addition, experimentation was performed in this research to understand if secondary MWF ingredients (other than so-called “extreme pressure” EP additives) might play a major role in affecting the tapping torque efficiencies observed for the base oil-in-water emulsions. In general, MWFs contain a large number of secondary ingredients that may include couplers (used to clarify the appearance of the MWF), corrosion inhibitors, chelating agents (to counteract the destabilizing impact of hard water ions), pH buffers, and others.

As formulated with secondary ingredients, a semi-synthetic MWF that has been investigated extensively in previous research has the following composition [5]: 57 wt% deionized water, 1.5 wt% coupler (butyl carbitol), 3.7 wt% tall oil fatty acid, 7.9 wt% corrosion inhibitor (monoethanol amine), 15 wt% oil, 14 wt% surfactants. As shown in Figure 6, it does not appear that the presence of these secondary ingredients has a significant impact on the tapping torque efficiency for the naphthenic based MWF base emulsion. As similar results have been observed by the authors for other MWFs, this suggests that the results in Figures 3-5 are general for the base fluids they represent. This, however, does not include the possible impact on tapping torque efficiency created by the presence of EP additives which were not investigated experimentally in this research. The potential role of EP additives in innovative MWF formulations is discussed in the next section.

Discussion: Impact of EP Additives on T^3

Extreme Pressure (EP) lubrication is a version of solid-film lubrication common to MWFs in which a solid-film forms through the corrosive action of EP additives under the extreme pressure and temperature conditions of cutting processes. As illustrated in Figure 7, EP additives used in MWFs are water insoluble organic chemicals (usually organo- phosphorus,

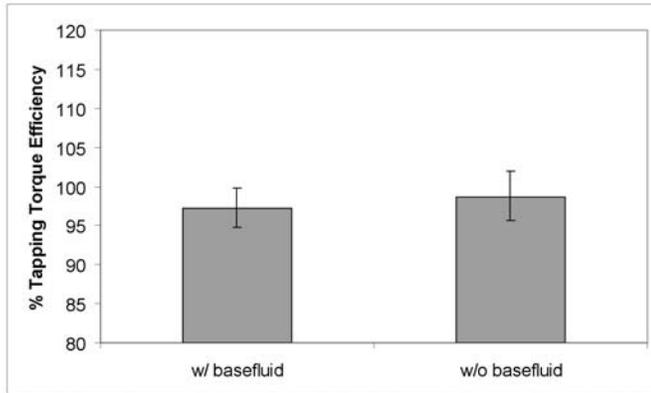


Figure 6. Tapping torque efficiency for naphthenic oil in water emulsions at semi-synthetic oil concentrations with and without secondary additives (butyl carbitol, fatty acid, monoethanolamine).

chlorine, or sulfur compounds) that decompose under extreme pressure-temperature conditions to form films of high melting point metal oxides and salts (of phosphate, chloride, and sulfide) on the tool and workpiece surfaces. These films have low shear stress and prevent direct metal-to-metal contact [11].

The choice of EP additives in a MWF depends on the temperature local to the cutting zone. Pressures in the cutting zone can be as high as 200 N/mm² for nonferrous metals and low-carbon steels to 1500 N/mm² for difficult-to-machine materials, and even higher for hardened steels [12]. In such cases, the temperature in the cutting zone can range from 350°C to above 1000°C due to the heat generated from metal deformation (in the metal shearing zone of the cut) and from friction between the chips and tools along the tool face as the cut metal is ejected from the cutting zone [11]. Since the mechanisms of EP additive effectiveness depends on the formation of metal oxides and salts, EP additive effectiveness depends on the local temperature. Generally, the temperature of EP additive activity for organo-phosphorous compounds ranges from 200-500°C. For organo-chloride compounds the range is 500-800°C, and for organo-sulfur compounds the range is 700-1000°C. As a single cut can span the full range of these temperatures as a function of the distance from the cutting zone, a suite of EP additives that span the whole range of temperatures may be found in a MWF formulation.

In order to be effective, EP additives must dissolve in the MWF formulation and reach the appropriate surfaces during the

cutting process. In semi-synthetic and soluble oil MWFs, the oil-in-water emulsion provides the needed hydrophobic host for the EP additives, since the nearly all the additives have limited solubility in water but very high solubility in oil. However, the EP additives must also get to the surface to produce effective lubrication films. Because semi-synthetic and soluble oil MWFs possess a variety of surface active emulsion stabilizing agents, an effective means for delivery is available. Specifically, the surfactants that stabilize the emulsion also produce organic films on the metal surfaces that provide a hydrophobic host for EP additives to partition into. EP delivery to the surface is thus accomplished by the transfer of the oil, EP additives, and other hydrophobic organic materials from the emulsion droplets to the organic films formed on the solid surfaces. More investigation is necessary to better understand the relationship between the selection of EP additives and their synergistic/antagonistic relationship with the balance of MWF formulations.

Results & Discussion: Impact of Particle Size on Lubricity

To avoid confounding the tapping torque efficiency results in Figures 3-5, the base emulsions in Figures 3-5 were not formulated with EP additives or secondary additives. Interestingly, while the soy and canola straight oils featured approximately the same tapping torque efficiency (Figure 3), the soy based MWF had slightly higher tapping torque efficiency in the semi-synthetic form (Figure 4). In the course of the research, it was questioned whether this might be due to the smaller particle size of the canola oil semi-synthetic (~0.6µm mean particle diameter) relative to the soy oil semi-synthetic (~1µm mean particle diameter). While it is known in metal forming operations such as rolling that such particle size considerations can be quite important, a similar investigation has not been described in the literature looking at this issue for a metal cutting operation such as tapping. Since anecdotal evidence has also suggested that emulsion droplet (or “particle”) size can also impact the bioresistance of a metalworking fluid, this issue was also investigated.

Figure 8 illustrates the relationship between increasing emulsion droplet (or “particle”) diameter, tapping torque performance, and microbial load for a naphthenic- semi-synthetic MWF formulation after the addition of calcium hydroxide [10]. A mean emulsion size shift from 20 to 2000 nm was observed and led to a slight, albeit statistically significant, improvement in tapping torque efficiency. Interestingly, the same emulsion particle size shift increased the total microbial load in the MWF by nearly 440%.

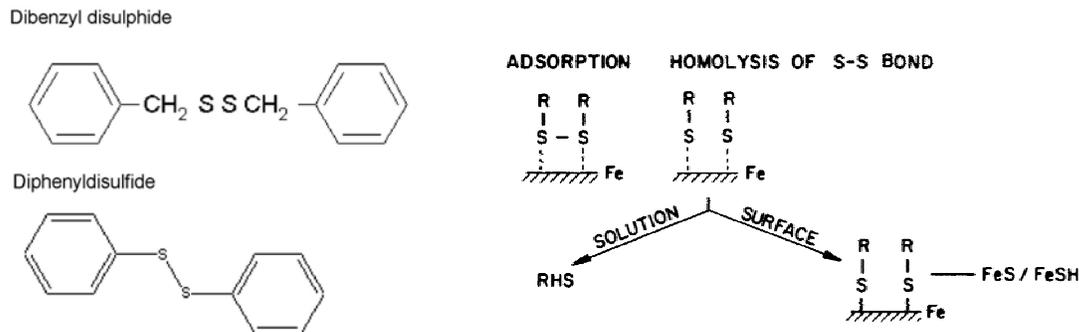


Figure 7. (Left) Two candidate organo-sulfur EP additives for MWFs: dibenzyl disulfide and diphenyldisulfide. (Right) Proposed mechanism of organo-sulfur action in metal cutting after Bushan (1999) [11].

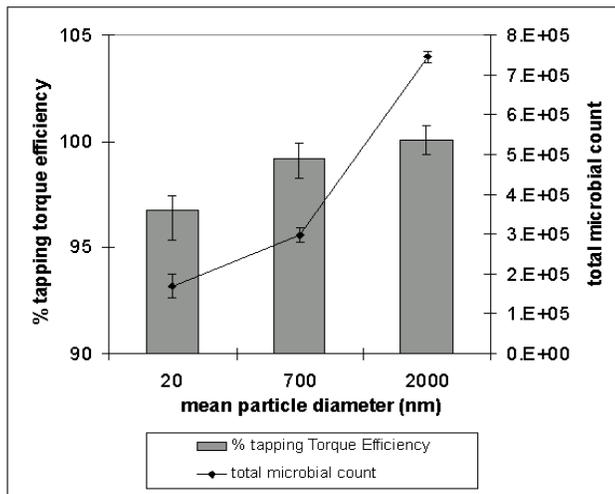


Figure 8. Tapping torque efficiency and microbial count as a function of mean particle size diameter for a naphthenic semi-synthetic MWF after Zimmerman et al. (2004) [10]

The reduction in bioresistance, and the increase of tapping torque efficiency, are likely to be due to the physical size of the MWF emulsion, rather than the addition of calcium. For the case of bioresistance, it is known that calcium is not typically a limiting nutrient in aqueous systems such as MWFs [13]. For the case of tapping torque efficiency, it is known that calcium contains no inherent lubricity characteristics. Particle size was shown to increase in the presence of other ions (e.g. magnesium) with similarly small increases in tapping torque efficiency [10]. Fluids at the same ion concentration had statistically identical tapping torque performance indicating that the final particle size determines the machining performance of the MWF regardless of the cause of the particle size shift. This suggests that particle size could be playing a role in the tapping torque efficiency differences observed between the straight oil and semi-synthetic MWF formulations for the soy versus canola base oils.

SUMMARY AND CONCLUSIONS

This study has used the tapping torque test to compare the performance of five base oil feedstocks for MWFs: naphthenic mineral oil, a 50/50 blend of naphthenic and paraffinic mineral oil, soybean oil, canola oil (75% oleic content), and a TMP Ester. The five oils were tested as straight oils, and formulated into soluble oil and semi-synthetic MWFs, to understand the impacts of emulsification on base oil performance. The results indicate that as straight oils, all vegetable based stocks, and the vegetable based ester, perform significantly better than the mineral oils. This trend holds, although is much less pronounced, after the vegetable stocks are emulsified into soluble oil and semi-synthetic MWFs. The results also indicate that some vegetable oil base stocks have a higher potential for lubricity than others, with data revealing that the soy and TMP ester provide improved tapping torque efficiency relative to canola oil in emulsified MWFs. Additional analysis suggests that such differences, although minor, may be related to the mean particle diameter of the MWF emulsions.

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