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REDUCING WASTE DISPOSAL OF METALWORKING FLUIDS BY ELECTRICAL IMPEDANCE MONITORING*

Marco Grossi, Bruno Riccò**

University "Alma Mater Studiorum" Bologna, DEI - Department of Electrical, Electronic and Information Engineering, Viale del Risorgimento 2, 40136 Bologna, Italy

Abstract

MetalWorking Fluids (MWFs) are widely used in mechanical industries for machine cooling and lubrication. Most MWFs are oil-in-water emulsions, with oil concentration ranging from 1% to 10%. In addition, anti-microbial compounds, corrosion inhibitors, emulsifiers, pressure additives and anti-foam agents are also normally added. MWFs degrade over time due to: growth of bacteria and fungi (that change the fluid pH); presence of contaminants (such as tramp oil); water characteristics (hardness, pH, high concentrations of chloride, sulphate and phosphate ions).
Once worn-out, the fluid becomes a waste to be properly disposed according to legislation and regulations, using techniques such as chemical waste treatment, evaporation, membrane filtration, electrocoagulation or biological treatment. The frequency of MWFs disposal should be as low as possible since this operation: a) represents a cost for both fluid disposal and replacement; b) has an impact on the environment. Thus, MWFs conditions should be regularly monitored and counteracting actions should be taken to make the product life as long as possible.
In this work we present a study on the possibility of monitoring MWF degradation by the analysis of the fluid electrical characteristics. Samples of MWFs (both fresh and degraded) have been analyzed measuring their impedance in the frequency range 20Hz to 2MHz. The acquired spectra were used in conjuction with Principal Component Analysis (PCA) for sample clustering and a model to estimate the fluids' pH, a key factor to assess wear-out, has been found that correlates well with values measured using the reference technique ($R^2 = 0.894$).

Keywords: fluid degradation, impedance spectroscopy, instrumentation, metalworking fluids, multivariate analysis, pH, sensors

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** Corresponding author: e-mail: marco.grossi8@unibo.it

1. Introduction

MetalWorking Fluids (MWFs) are products widely used in mechanical industries to cool and lubricate both worked pieces and machine tools during different metal finishing processes (Stephenson and Agapiou, 2005). MWFs can be classified in three main groups according to their composition: mineral oils (petroleum based), semi-sinthetic oils (obtained by emulsion or microemulsion of mineral oil with water) and synthetic oils (based on alkaline compounds). Water based MWFs contain oil concentration in the range 1% to 10% depending on the specific product and the type of material to be worked. In addition they also contain anti-microbial compounds, corrosion inhibitors, pressure additives and anti-foam agents.

MWFs degrade over time with use due to different causes, among which one of the most important is microorganism growth (Bakalova et al., 2007; Mattsby-Baltzer et al., 1989). Bacteria and fungi can grow in most MWFs products (especially the water-based ones) due to the nutrients present in the emulsion, such as glycols, fatty acid soaps and amines. Microorganism growth makes the fluid become "rancid", with bad smell (in particular in presence of sulphate reducing bacteria), decrease in lubricating properties, increase of corrosion of both machine tools and worked pieces, reduction of production quality and higher probability of machines malfunction. High concentration of microorganisms ($> 10^7$ cfu/mL) results in a decrease of the fluid pH (due to microbial metabolism) that, in turn, makes the environment more favorable to further microbial growth.

To counteract this process, a biocide is often added to the fluid at regular intervals to decrease the microbial concentration and restore the fluid pH at a level (about 9.0) where microbial growth is very slow. While biocides are effective in contrasting microbial growth (Marchand et al., 2010), problems are in order concerning toxicity (most of biocides can release formaldehyde, a well known irritant for the respiratory tract and a carcinogenic agent) and potential generation of biocide-resistant microorganism strains. Since during use MWFs are dispersed in the air in the form of aerosol, studies have shown how respiratory problems of exposed workers increase with higher concentrations of MWF mists (Kriebel et al., 1997; Zacharisen et al., 1998). And even if MWF mist concentration is within legal standard, 20% of workers have been found to suffer from work-related respiratory problems (Rosenman et al., 1997).

To optimize MWFs performance and extend their lifetime it is important to maintain the proper oil concentration, which varies with time due to water evaporation, bacterial attack, oil adhesion to metal parts, etc. The industrial standard for oil concentration measurement is refractometry (Canter, 2011), a technique that allows quick and in-situ measurements but is strongly affected by fluid contamination. Alternative approaches are based on the measurements of viscosity (Grossi et al., 2016), density (Navarro de Andrade et al., 1999) and ultrasound speed (Meyer and Saiz-Jabardo, 1994).

Another factor that contributes to the degradation of MWFs is contamination. MWFs are often contaminated by metal particles with composition that varies widely with cutting process and operating conditions. Another source of contamination is non-soluble oil (tramp oil) leaking into the MWFs tank (Rakic and Rakic, 2002). Contamination has negative effect on corrosion inhibition, while lubrication and cooling are negatively affected only if contamination is so high to affect the emulsion stability (Greeley and Rajagopalan, 2004).

Once degraded, MWFs become a waste and must be disposed according to legislation and regulations. The first step in the disposal process is the separation of oil and water, that can be achieved by means of conventional techniques (such as gravity separation, dissolved air flotation and de-emulsification) or with the more recent membrane technology (Cheryan and Rajagopalan, 1998; Hesampour et al., 2008) that includes microfiltration, ultrafiltration, nanofiltration and reverse osmosis. Other techniques for MWFs disposal are

electrocoagulation (Kobya et al., 2008) or biological treatment by means of inoculum of bacteria to degrade the chemical constituents and additives present in the fluid (Van Der Gast et al., 2004).

MWFs disposal represents a serious problem from both a monetary and an environmental point of view. Thus the fluid lifetime must be extended as long as possible.

In this paper we present a technique, based on Electrical Impedance Spectroscopy (EIS), to monitor the degradation of water based MWF samples. EIS is a powerful techniques that is based on the measurement of electrical properties of the material of interest in a wide range of frequencies and has been used for sensing applications in wide range of different fields, such as: quick detection of microbial concentration (Choi et al., 2009; Grossi et al., 2008; Grossi et al., 2009; Grossi et al., 2010; Grossi et al., 2011a; Grossi et al., 2012a; Grossi et al., 2013a; Grossi et al., 2013b; Grossi et al. 2014a; Hardy et al., 1977; Johnson et al., 2014; Mancuso et al., 2016; Pompei et al., 2012; Puttaswamy and Sengupta, 2010; Settu et al., 2015; Uria et al., 2016; Wang et al., 2012) analysis of human body composition (Gudivaka et al., 1999; Ibrahim et al., 2005; Kyle et al., 2001; Rush et al., 2006); the characterization of food products (Ferrero et al., 2014; Grossi et al., 2011b; Grossi et al., 2012b; Grossi et al., 2013c; Grossi et al., 2014b; Grossi et al., 2014c; Jackson et al., 2000; Yang et al., 2016); study of the degradation of organic coatings for metallic surfaces in contact with acid electrolytes (Bonora et al., 1995; Loveday et al., 2004).

For this work, 10 MWFs samples (both fresh and contaminated) have been studied in the frequency range 20Hz – 2MHz using a LCR meter Agilent E4980A. Acquired impedance data have been modeled using an equivalent electrical circuit and processed using multivariate data analysis. The results have shown that: a) it is possible to discriminate between fresh and contaminated samples; b) the fluid pH can be estimated with good accuracy.

The possibility to monitor the fluid degradation by non-destructive electrical measurements (that can be done directly in fluids' tanks, even remotely) is very important since it allows on-line monitoring of MWFs without the need to ship samples to a laboratory, thus resulting in lower costs and lower time response for the analysis. This, in turn, provides the possibility to extend MWFs lifetime, with less costs for fluid replacement and benefits for the environment.

2. Materials and methods

All Samples Under Test (SUT) used in this work are oil-in-water emulsions (both fresh and contaminated) with oil concentration in the range 1% to 10%. Ten different MWFs have been tested: SUTs #1, #2 and #3 are fresh MWFs created mixing a soluble oil produced by Total with tap water in different oil concentrations (2% for #1, 6% for #2 and 10% for #3, respectively). SUTs #4, #5 and #6, instead, are MWFs used for some time in a metalworking plant and are characterized by a moderate level of contamination. SUTs #7, #8, #9 and #10 are MWFs used for a long time and strongly contaminated. For each sample, the pH was measured using a portable pH meter (HI 9811-5N) and the obtained values have been used as reference for this work.

All experiments have been done using the set-up shown in Fig. 1 (a). A 50mL Falcon vial (hereafter called "the sensor") was modified to feature a couple of stainless steel electrodes (diameter 6mm, 12mm apart) to be used for electrical measurements, done by means of an Agilent E4980A LCR meter. A sine-wave voltage signal of amplitude 100mV and frequency ranging from 20Hz to 2MHz has been applied to the sensor electrodes measuring the complex impedance Z, with its real and imaginary components (Re(Z) and Im(Z), respectively). The data are transferred to a lap-top for data displaying, filing and further analysis.

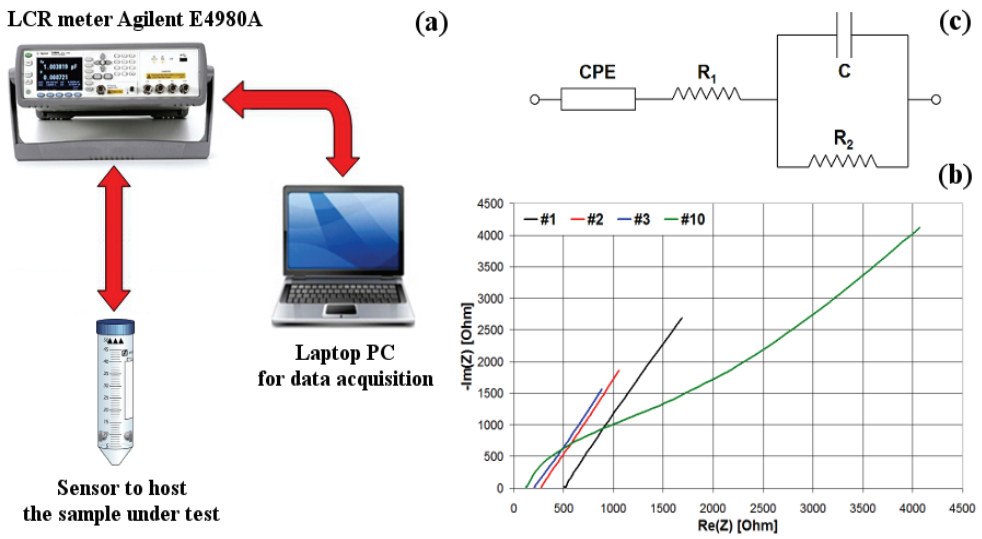

Fig. 1. Measurement setup used in this work (a). Nyquist plot for four different tested samples (b). Equivalent electrical circuit for the sample-electrode system (c)

Fig. 1 (b) shows the Nyquist plot (-Im(Z) vs. Re(Z)) for all frequencies in the case of four different samples, three fresh and one contaminated. As can be seen, while the fresh samples (#1, #2 and #3) are represented by a straight line, the contaminated SUT (#10) exhibits a more complex behavior with lower values of Re(Z) at high frequencies and higher values for both Re(Z) and –Im(Z) at low frequencies.

The measured impedance data have been modeled with the equivalent circuit shown in Fig. 1 (c): the MWF is essentially modeled by means of the resistances R_1 and R_2 and the capacitance C, while the Constant Phase Element (CPE) accounts for the non-linear electrode-electrolyte interfaces.

CPE is a non-linear circuit element whose impedance can be described by two parameters (Q and α) with Eq. (1):

$$Z_{CPE} = \frac{1}{Q \cdot (j\omega)^{\alpha}} \tag{1}$$

where Q represents the double-layer capacitance, while α accounts for the non-ideal electrodes-electrolyte interface (with α = 1 indicating an ideal capacitance).

The measured impedance spectra have been fitted to the electrical circuit of Fig. 1 (c) using the software Multiple Electrochemical Impedance Spectra Parametrization (MEISP) v3.0, by Kumho Chemical Laboratories and the values of the electrical parameters have been worked out.

Fig. 2 shows the Bode plots (Re(Z) and -Im(Z) vs frequency) for both measured and fitted data in the case of sample #10 and, as can be seen, the model fits very well the measured data. Since pH is a very important parameter used to monitor MWF contamination, for each SUT it was measured using a 9811-5N pH meter by Hanna Instruments.

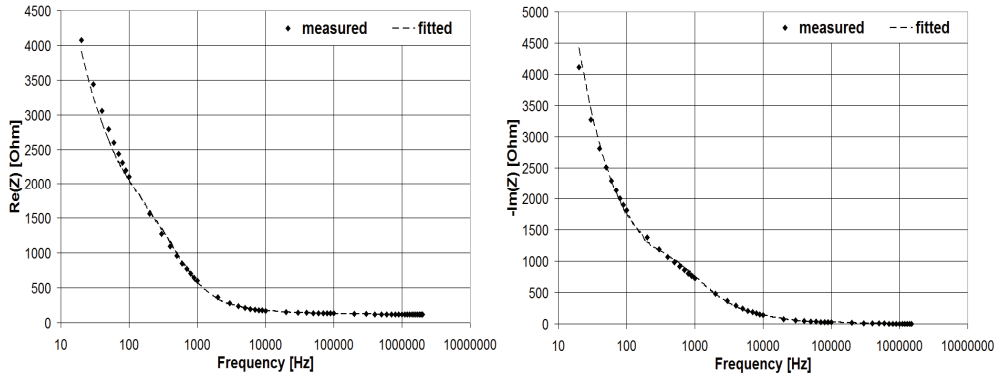

Fig. 2. Bode plot, of Re(Z) and –Im(Z) vs. frequency, in the case of SUT #10.

All the statistical analysis have been carried out using Microsoft Excel and its statistical package XLSTAT (by Addinsoft).

3. Results and discussion

The electrical parameters from the equivalent model of Fig. 1 (c) and the measured pH values are presented in Table 1 for all the SUTs.

Table 1. Electrical parameters fitting the equivalent circuit and pH values from the measured samples

Samples	*pH*	*Q (μF)*	*α*	*R_1 (Ω)*	*R_2 (Ω)*	*C (μF)*
#1	9.1	8.807	0.7544	438.85	74.75	0.0004362
#2	9.17	12.648	0.7594	271.73	37.79	24.96
#3	9.24	16.134	0.7433	202.65	24.57	20.96
#4	8.4	3.136	0.7606	166.47	5936.3	0.3404
#5	8.1	8.722	0.6523	197.2	2932	0.2695
#6	7.8	7.981	0.684	155.98	1067.5	1.198
#7	7.56	7.445	0.5903	142.34	4646.1	0.04682
#8	7.6	6.798	0.6201	200.1	3667.7	0.1037
#9	7.6	8.953	0.6296	134.34	1777.4	0.1432
#10	7.7	9.104	0.6293	116.43	907.2	0.3138

As can be seen, the pH value is clearly correlated with the fluid contamination level. The fresh samples (#1 to #3) are characterized by high pH values (from 9.1 to 9.24) because of the alkaline compounds of the oil from which they were produced. As the samples become more and more contaminated, the pH value gradually decreases, mainly because of the bacterial metabolic activity producing acidification. This is shown in Table 1, where contaminated samples (#4 to #6) exhibit lower pH values than fresh ones (7.8 to 8.4), while highly contaminated SUTs (#7 to #10) feature even lower pH values (7.56 to 7.7).

Regarding the system equivalent model, R_1 and R_2 are the parameters most influenced by the fluid contamination: R_1 is higher for fresh samples and decreases for contaminated ones, while for R_2 it is the opposite.

In both cases, however, the sample pH cannot be estimated with good accuracy from these parameters. As shown in Fig. 3 the correlation between the sample pH and R_1 or R_2 is rather low ($R^2 = 0.4906$ for R_1 and $R^2 = 0.2542$ for R_2).

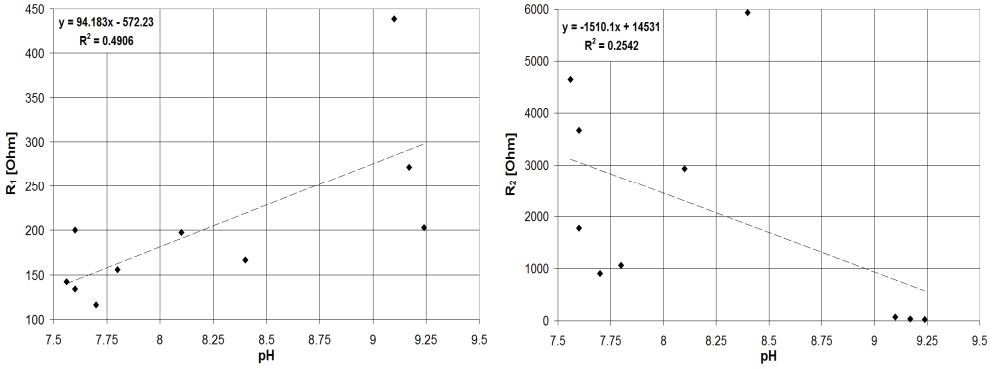

Fig. 3. Scatter plot of the electrical parameters R_1 and R_2 plotted versus the measured pH value

To improve the accuracy in sample classification, a multivariate data analysis has been implemented on the full acquired impedance spectrum: for each SUT, the data of Re(Z) and Im(Z) for every frequency have been used with a Principal Component Analysis (PCA) algorithm to obtain a set of linearly uncorrelated variables. The first two principal components (F1 and F2) are responsible for more than 98% of the whole spectrum variations and Fig. 4 shows a scatter plot of F1 and F2 for all tested samples.

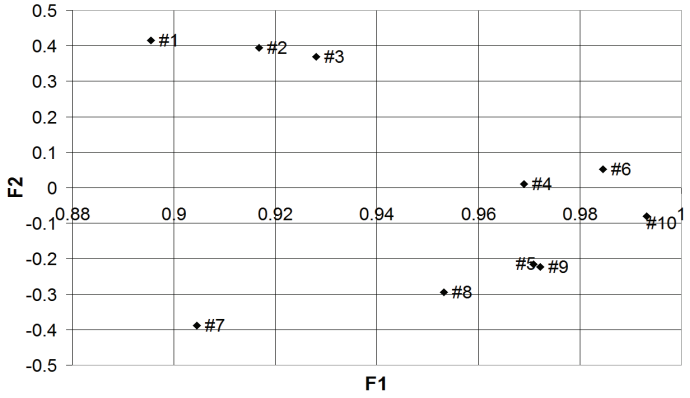

Fig. 4. Scatter plot of the first two principal components for all the tested samples

As can be seen the fresh uncontaminated samples (in the higher left portion of the plot) are strongly separated from the remaining contaminated SUTs. In particular, the second principal component F2 can effectively discriminate fresh samples (F2 > 0.2) from contaminated ones (F2 < 0.2). The discrimination between moderately (#4 to #6) and highly contaminated samples (#7 to #10), instead is more problematic, although the former ones are in general characterized by higher values of both F1 and F2 than the latter ones.

The possibility to reliably discriminate the three groups of samples has been investigated analyzing the results from PCA with two different clustering algorithms, Factorial Discriminant Analysis (FDA) and k-means clustering, and the results are presented in Table 2.

Table 2. Clustering of the investigated samples using FDA and k-means algorithm

Samples	*Group*	*Result from FDA*	*Result from k-means algorithm*
#1	Not contaminated	Not contaminated	Not contaminated
#2	Not contaminated	Not contaminated	Not contaminated
#3	Not contaminated	Not contaminated	Not contaminated
#4	Contaminated	Contaminated	Contaminated
#5	Contaminated	Highly contaminated	Highly contaminated
#6	Contaminated	Contaminated	Contaminated
#7	Highly contaminated	Highly contaminated	Highly contaminated
#8	Highly contaminated	Highly contaminated	Highly contaminated
#9	Highly contaminated	Highly contaminated	Highly contaminated
#10	Highly contaminated	Contaminated	Contaminated

Both these algorithms give the same results: fresh samples are always correctly clustered, while contaminated and highly contaminated SUTs are casted correctly with probability 66.6% and 75%, respectively.

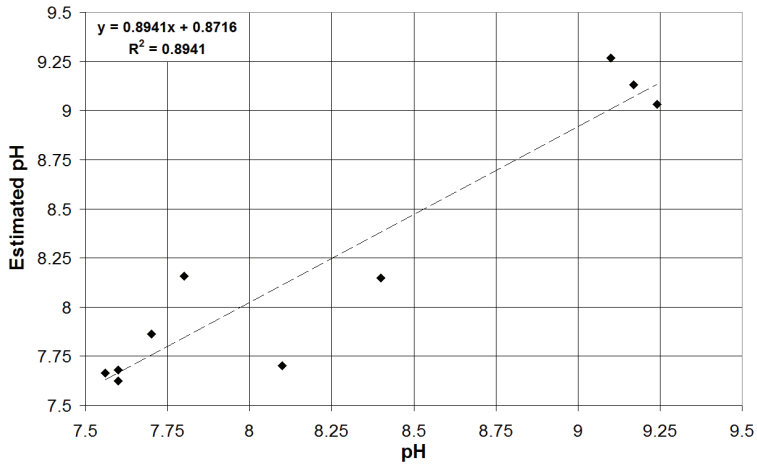

Fig. 5. Scatter plot of the estimated pH versus the value measured with a pH-meter

Finally, the output data of PCA (F1 and F2) have been used to estimate the pH value of the samples under test using Multiple Linear Regression (MLR). The regression line best fitting the experimental data was found to be:

$$pH = 12.5169 - 4.5298 \times F1 + 1.944 \times F2 \tag{2}$$

The scatter plot of the pH values estimated by means of Eq. 2 vs. those measured with the pH-meter is represented in Fig. 5. As can be seen, the use of PCA in conjunction with MLR results in much better accuracy for the estimated pH ($R^2 = 0.8941$) compared with the results obtained using the fitted electrical parameter R_1 or R_2.

4. Concluding remarks

In this work a novel technique has been presented to monitor the degradation of water based MetalWorking Fluids (MWF). The technique is based on impedance measurement of the sample under test on a wide range of frequencies (20Hz – 2MHz). The measured

impedance data are analyzed using Principal Component Analysis (PCA) to extract a set of two linear uncorrelated variables (principal components) that describes more than 98% of the full spectrum variations. The two principal components are then satisfactorily used to discriminate between fresh and contaminated samples and to estimate the sample pH value.

The proposed technique can thus be used for on-line and in-situ monitoring of MWFs degradation. This, in turn, allows quick counteracting actions when the fluid contamination exceeds some critical threshold resulting in longer fluid lifetime, lower costs for product disposal and replacement. Hence ultimately also in less impact on the environment.

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