Influence of 4-dodecylbenzenesulfonic acid as a surfactant on a graphite-based dielectric in powder mixed electric discharge machining

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Influence of 4-dodecylbenzenesulfonic acid as a surfactant on a graphite-based dielectric in powder mixed electric discharge machining

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Abstract

A recent trend in micro EDM is the addition of powders into the dielectric. The presence of powders helps to lower the dielectric breakdown voltage and therefore the discharge occurs early. As a result, the discharge energy is better distributed, resulting in a greater number of discharges, each with less energy. The main advantage of using this method is the improvement of both the process performance and surface finishing of the workpiece. A critical aspect of the implementation of this technology is in general the management of the powder. In fact, to obtain advantages during the machining, the powders should be maintained dispersed into the dielectric to avoid their aggregation. This paper aims to study the concentration of the powder and the surfactant in the dielectric fluid on micro EDM drilling performance. Titanium alloy was used as workpiece material, hydrocarbon oil as dielectric, graphite as powder and 4-dodecylbenzenesulfonic acid as surfactant. The performance was evaluated considering the Material Removal Rate, the Tool Wear Ratio and the geometrical characteristics of the holes (overcut and taper rate). Graphite content positively affected both Material Removal Rate and Tool Wear Ratio; a larger spark gap was observed as well. The use of surfactant is required for mix stability but increasing its percentage generally reduces the effects of graphite and increases data dispersion as well.

Keywords: micro-EDM, micro-drilling, PMEDM, graphite, surfactant

1. Introduction

EDM (Electrical Discharge Machining) is a removal process in which the material is eroded by electrical discharges between the electrode tool and the workpiece. There is a dielectric fluid between the tool and the workpiece, which electrically isolates the workpiece from the electrode and permits the discharge to take place only when the potential difference exceeds the breaking voltage of the dielectric fluid.

The process is not influenced by the mechanical properties of the workpiece material. For this reason, it is suited to machine electrically conductive materials that are difficult to process using conventional technologies due to their mechanical properties.

The material removal process takes place through the formation of craters, each of which is removed by a single discharge. The dimension of the craters is very small, guarantying very accurate machining. Anyway, a limit of this technology is represented by the low material removal rate. One of the last tendencies in the research of EDM, to improve its performance, is the addition of micro/nano particles in the dielectric fluid. In this case, the technology is named PMEDM (Powder Mixed Electrical Discharge Machining). Using powders helps lower the dielectric breakdown voltage; therefore, the discharge occurs early. A bridge in the gap between workpiece and electrode is formed and when an electrical discharge occurs, the bridge breaks up and the powder particles are dispersed within the workpiece-tool gap. This phenomenon permits a more uniform distribution of the discharge energy, resulting in a greater number of discharges, each with less energy. The breakdown voltage decreases, the discharge frequency increases and finally the material removal occurs faster [1]. The energy is distributed among the electrode and the workpiece more effectively, producing smaller diameter craters and also improving the surface finish [2].

The size of the particles is a significant parameter, and its selection mainly depends on the application, EDM or micro EDM [3]. Other parameters related to the powder are the electrical conductivity and the concentration. The powders can be conductive, semi-conductive or not conductive. Among the conductive powders, the most commonly used is graphite, considered a good electrical conductor. Other conductive powders such as aluminium and carbon nanotubes are also reported. Less commonly used but still worth mentioning are powders of titanium, tungsten, manganese, and other metals.

Among the semi-conductive powders, silicon carbide and boron carbide are the most studied in EDM. However, no electrically conductive powders are also taken into account, like boron oxide or aluminium oxide [4, 5]. In this case, the aim of the powders is the modification of the surface properties of the workpiece, the abrasion and wear resistance of the material improves. Anyway, using these types of powder causes a larger working gap and the discharges become more uniform, permitting an optimal surface finishing and a minimum depth of the white layer.

The concentration is another important parameter related to the PMEDM. Several papers are available in the literature in which the optimal powder concentration in the liquid was found [6-10].

Generally, the performance of the EDM process is evaluated using some indexes. For example, MRR (Material Removal Rate) and EWR (Electrode Wear Rate) are both expressed in mm^3/min, while TWR (Tool Wear Ratio) is calculated as the ratio between the volume of material removed from the electrode and that removed from the electrode. The machining is also evaluated considering geometrical aspects such as in the case of drilling operation the overcut and the taper rate. Finally, the properties of the workpiece in terms of hardness increase after machining are also taken into account.

In general, powder mixed EDM offers several advantages. First of all, the performance is better: both MRR and TWR improve compared with the traditional EDM. In [11] and [12], the researchers investigated the advantages introduced by the use of graphite powder in the dielectric by observing how it affects both MRR and TWR in die-sinking. MRR improves significantly (from 25 to 143% as a function of peak current and pulse o time) and TWR decreases moderately. An optimal value of the graphite concentration was found, equal to 5 g/l. Similar results were found in [13] where graphite was added to kerosene: machining time was decreased by about 35%. In [14] the MRR improves by around 74% and TWR decreases by 94%. In [15] it was found that the MRR as a function of the powder concentration shows a maximum; if the powder concentration is higher than this value the performance gets worst. Moreover, the optimal powder concentration is not the same for all the types of powder but depends on the powder.

The properties of the type of powder added to the dielectric fluid influence the performance. A comparison between two types of powders was made in [16], where JIS SKD11 was drilled using graphite and boron carbide (B4C) in the dielectric. The improvement in MRR is greater using graphite than boron carbide, probably due to the higher electrical conductivity of the graphite. However, the electrode wear is subjected to a bigger erosion.
The effect of the electrical conductivity of the powder in the dielectric is also confirmed in [17].
In [18] the size of the powders was taken into account. Its effects on the machining time seem to be negligible while, in general, decreasing the diameter of the particles the length of the worn electrode slightly decreases.

Regarding the quality aspects, the width and depth of the craters using powders in the dielectric are smaller; therefore, the surface finishing improves thanks to a higher number of sparks having lower energy [13, 16]. In [10] the authors estimated that the roughness surface decreases in the range between 2.4 and 18.3% as a function of the process parameters. There is an optimal value of graphite concentration to improve the surface finish [11].
In general the geometrical aspects, taper rate and overcut get worst using PMEDM and are influenced by the powder concentration [17].

As far as the surface topography, EDM is a thermal technology and an altered thermal zone is unavoidable, where thermal stresses and microcracks are possible. Using PMEDM these phenomena undergo a marked reduction [15]. Considering the lower power density of the energy sparks than EDM traditional, the micro-cracks on the surface are reported to be less (for traditional EDM the density is 0.03 cm^{-1} while with PMEDM is 0.004 cm^{-1}) [13].

The microhardness of the machined workpiece in general increases after EDM machining but in the case of PMEDM the increase is lower than traditional EDM [19]. In [1] the authors found that the concentration of graphite in the dielectric affects the microhardness value.

The use of powders in the dielectric may change the chemical composition of the workpiece surface. In fact, the migration of the powder from the dielectric to the workpiece produces new chemical phases as a function of the powder type [16, 20]. As a result, in some cases, the electrical chemical corrosion resistance of the workpiece material may improve, and the electrical conductivity may increase.

An important task that often is not deeply discussed is mixing powders in the dielectric. Mechanical systems like stirrers are usually fixed to the EDM tank in order to avoid the natural deposition of the powders on the bottom of the tank [21]. In some cases, a circulating pump is used to assure dielectric circulation [22-24]. These solutions guarantee the presence of the powder in all the dielectric but are not able to avoid the aggregation of some powder particles. For this purpose, it is necessary to use additives in the dielectric dispersion like a surfactant [25-26]. In fact, compared to standard EDM, in micro EDM the particle size is especially important. Because of the smaller gap between electrode and workpiece, large particles may likely lead to short circuits and thus poor process performance [3].
This work focuses on studying the benefits of both the powder and the surfactant in the dielectric liquid. As already said, the main role of the surfactant is the prevent aggregation of the powder

particles. The amount of surfactant in the solution is strictly connected to the concentration of powder that is used.

The aim of the paper is the study the effects of both powder and surfactant concentrations in the dielectric on the micro EDM drilling performance. Titanium sheets having a thickness of 0.5mm were drilled using a WC cylindrical electrode having a diameter of 0.1mm. Graphite and 4-dodecylbenzenesulfonic acid were used at different concentrations as powder and as a surfactant, respectively. The process was evaluated using both performance and geometrical indexes such as Material Removal Rate (MRR), Tool Wear Ratio (TWR), Diametral Over Cut (DOC) and Taper Rate (TR). In this way, the solution optimizing the benefits of the powders in PMEDM was found.

2. Experimental plan

An experimental campaign was carried out by drilling micro-holes on a titanium sheet (Ti6Al4V) having 0.5mm thickness. Tungsten carbide electrodes of 0.1mm diameter were used. The tests were carried out using a µEDM machine, Sarix SX-200.

The same finishing process parameters were used during the campaign, as reported in Table 1. Gain is the parameter that controls the gain of the reaction block; the gap is a value proportional to the distance between the electrode and the workpiece during the erosion; energy defines the shape of the pulse; regulation identifies a certain regulation management algorithm defined by the machine manufacturer.

Table 1 EDM process parameters

Polarity	negative
Peak current [index]	90
Voltage [V]	80
Width [µs]	1
Frequency [kHz]	160
Energy	13
Gap	70
Gain	50
Regulation	00-00

Hydrocarbon oil was used as base dielectric. As powder, graphite (C>99.9%, density 1.9 g/cm^3) having a diameter between 10 and 30 nm was added into the dielectric, varying its concentration.

4-dodecylbenzenesulfonic acid (DBSA, mixture of isomers ≥ 95%, Sigma Aldrich) was used as a surfactant for graphite dispersion. DBSA was added at different concentrations into the dielectric fluid. The graphite/DBSA oil mixtures were stirred for 30 minutes and then sonicated for 30 minutes before all experimental tests to ensure uniform graphite distribution. Thus, the homogenously mixed dielectric fluid was added to the machine.

Tests varying the concentration of both graphite and surfactant were made. Selected surfactant concentrations were investigated to obtain the best graphite dispersion, thus increasing the dispersion stability according to the absorption process that depends on the magnitude of the hydrophobic bonding free energy [27]. Fig 1 shows the experimental plan. As a starting point, the maximum graphite concentration was fixed at 3g/l with a surfactant concentration of 10% in volume. Beyond this point, it is difficult to prevent the fast separation of the graphite in the liquid. Therefore, the first set of experiments was planned by keeping constant the ratio between graphite and surfactant (red points) by reducing the concentration of both additives. In another set of tests, the surfactant concentration was kept constant (10%), and the graphite concentration was varied (yellow points). Last, another group of tests was aimed to evaluate intermediate conditions (green points). The shaded area in Fig 1 corresponds to a combination having a higher concentration of graphite concerning surfactant leading to unstable graphite in the liquid. For this reason, this region was not tested.

It is worth noting that in some experimental conditions no graphite was added. In particular, pure hydrocarbon oil (the origin of the graph in figure 1) was tested as a reference condition. Furthermore, in one of the experimental conditions only surfactant was added to hydrocarbon oil.

For each experimental condition, at least two series of 10 micro-holes were executed.

Different ratios between Graphite [g/l] and surfactant [%Vol] are labelled using a value, that has been reported in all subsequent figures as a dimensionless number (i.e. 0.3, 0.15 etc.).

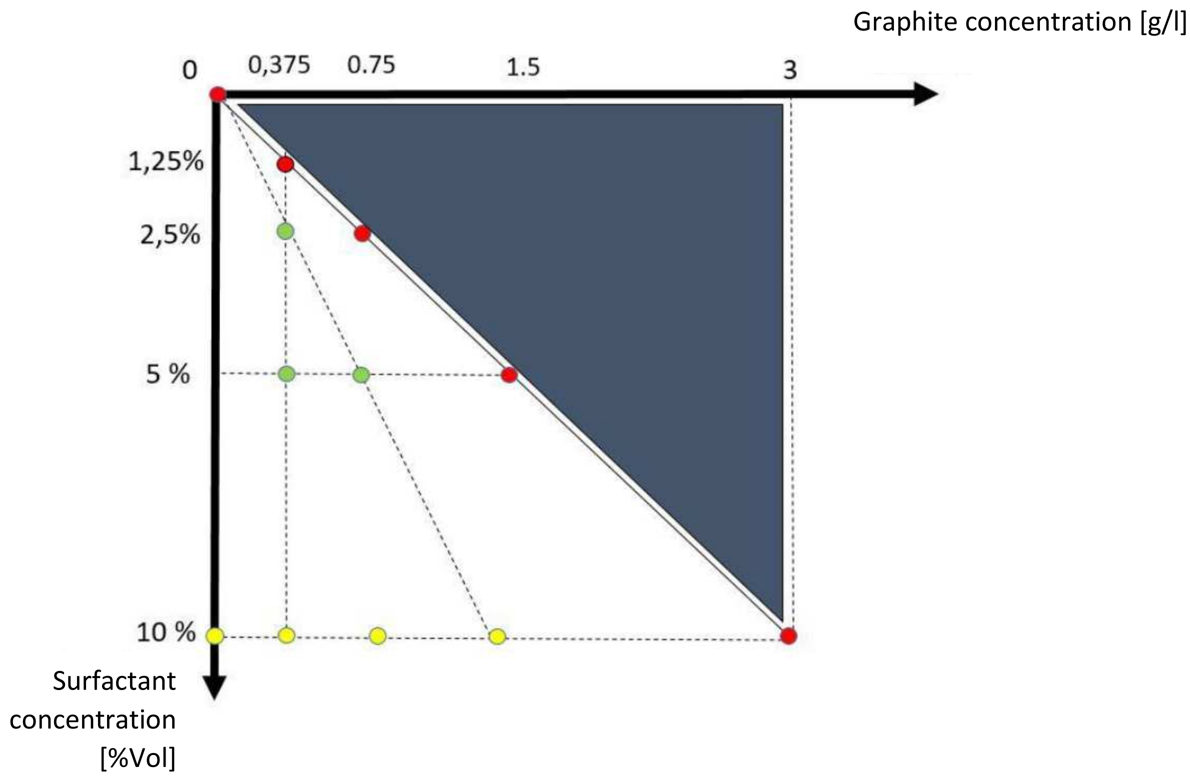

Fig. 1 Experimental plan

Micro-holes were realized using an automated programme that measures the machining time of each hole and the frontal electrode wear through a touching operation in a reference point. The wear is calculated as the difference between the start length of the electrode and the final length after the machining operation.

The geometrical characterization of the micro holes was made using an optical microscope: the top and bottom diameters were measured.

The performance of the machining was evaluated considering several indexes: machining time expressed in seconds [s], electrode wear in [mm], MRR (Material Removal Rate) in [mm^3/min], TWR (Tool Wear Ratio) calculated as the ratio between material volume removed to the electrode and to the workpiece, DOC (Diametral Over Cut) calculated as the difference between the hole top diameter and the electrode diameter expressed in [µm] and TR (Taper Rate) calculated as the ratio between the difference of top and bottom diameter and the hole thickness, that represents the conicity of the hole. The results are discussed in the following section.

3. Analysis of the results

The performance was evaluated taking into account both indexes related to the process and the geometrical characterization of the holes. In the following figures, each point represents the average value over the series of 10 consecutive holes.
The effects of the graphite concentration on the machining time-varying the ratio between graphite and surfactant are reported in Fig 2.
The yellow points represent the results obtained with pure dielectric while the green points that with adding only surfactant in the dielectric. First, by adding surfactant in a pure dielectric the machining time worsens. In fact, the effect of surfactant is to remove the carbon particles that are formed due to the high spark energy in the hydrocarbon oils. The presence of the carbon particles helps the machining act like the added powders and therefore the surfactant significantly reduces carbon's contribution (compare yellow and green points) [28].
The presence of graphite affects the machining time, especially when its concentration is high. For low graphite concentration, this effect is less clear. In fact, in these conditions, the benefits of the presence of surfactant may be negative, subtracting the poor graphite powders from the machining zone. The ratio between the graphite and surfactant concentration seems to be a critical aspect. The main role of the surfactant is to avoid the agglomeration and precipitation of the powders.
DBSA was selected for the dual-action induced from its molecular structure bearing a 12-carbon alkyl chain and a benzene ring (as a hydrophobic segment), as well as an SO_3^- charged head group (as a hydrophilic segment).
The long alkyl chain could be easily absorbed on the graphite surface, also enhanced by π-stacking interactions of benzene and delocalized π-electron of graphite [29], thus improving the surface coverage degree of graphite [30]. On the other side, SO_3^- groups strengthen the electrostatic repulsion between DBSA-covered graphite due to screened Coulomb interactions, thus stabilizing the surfactant adsorbed onto particles and avoiding particle aggregations also thanks to the presence of the long alkyl chain [31].
Moreover, as reported in the literature, the simultaneous use of aromatic surfactant and open-chain aliphatic hydrocarbon dielectric fluid significantly influences the ignition and breakdown phase of a single discharge [27]. Indeed, non-polar groups and aromatic structures lead to average fast ignition at lower voltages. Accordingly, aromatic surfactants are easily dispersed in dielectric oil and can react strongly to electron transfer reactions.
Furthermore, electrical barriers produced by surfactant molecules (lyophilic chains) can stabilize the dispersion of graphite and surfactant in the dielectric fluids, thanks to their absorption on the

surface of the graphite surface and the interaction among powder particles, surfactant and dielectric.

Under the ratio of 0.3, the dispersion is unstable, and the powders get down in a few minutes, making the mixing unsuitable for use. Anyway, it is not useful to adopt a high-value surfactant because the machining time does not improve, dielectric costs increase and finally makes all process less reproducible and sustainable. In these tests, the optimal ratio between the graphite and surfactant concentration is 0.3.

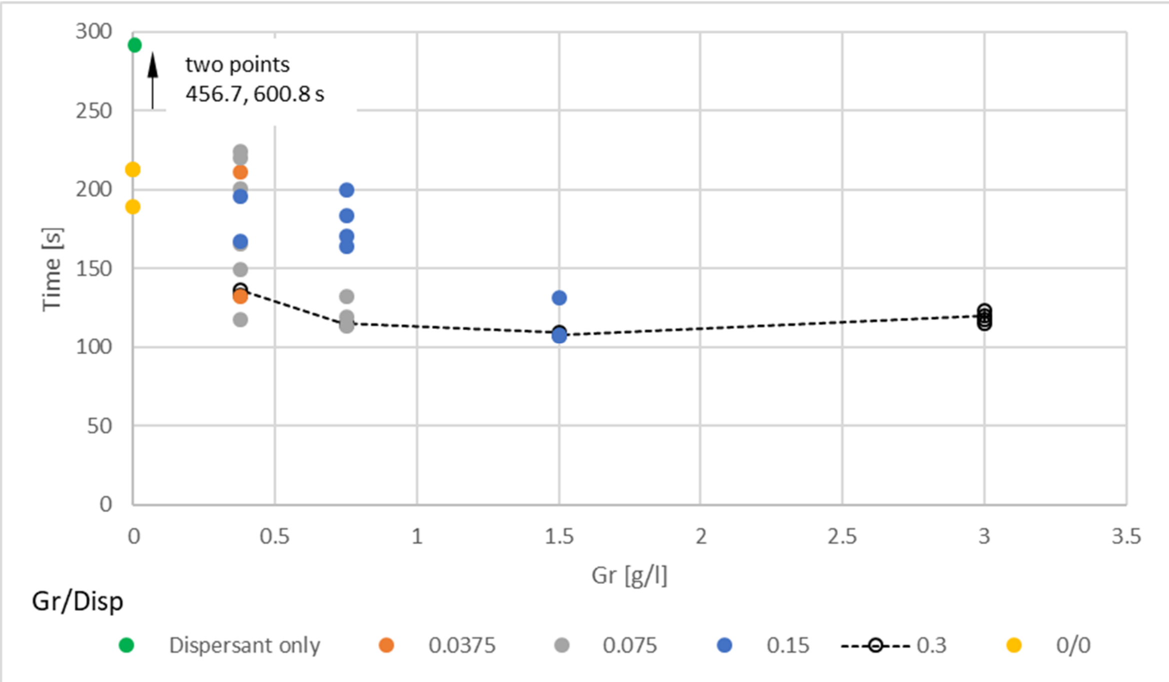

Fig. 2 Machining time as a function of the graphite concentration varying the ratio between graphite and surfactant

From Fig 3, it can be noted that the wear adopting no graphite is among the lowest results. This is especially true when surfactant is used regardless of exceptionally high machining time. This result is strange since graphite should protect the electrode. A possible explanation may involve the effects of the graphite originating from hydrocarbon oil during the sparks. Furthermore, when DBSA is present, graphite may be active for a short time before the action of the surfactant occurs possibly due to its slower kinetic. Anyway, the difference between the maximum and minimum wear is very small, around 20 µm.

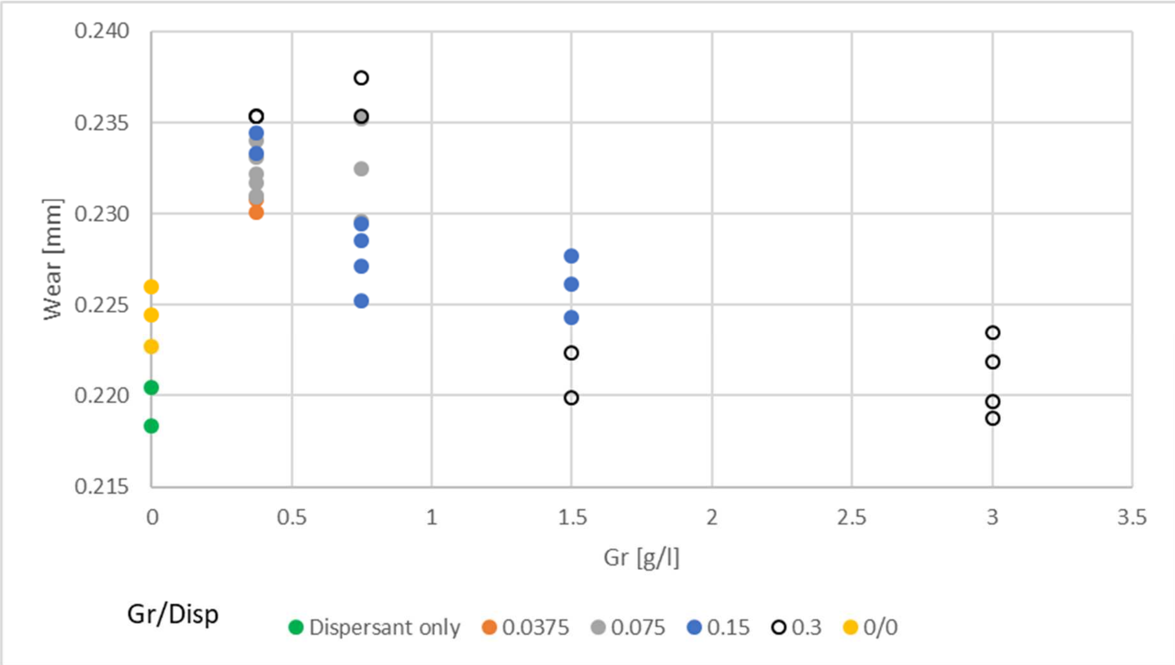

Fig. 3 Electrode frontal wear as a function of the graphite concentration varying the ratio between graphite and surfactant

The considerations made for machining time can be extended to MRR (Fig. 4). The worst performance occurs when only the surfactant is added to the dielectric: in this case, the MRR gets worse at around 60-70% than in the traditional condition. The graphite has effects on MRR and there seems to be a light trend with increasing MRR as the powder concentration increases. The use of a quantity of surfactant should be made accurately: low values of surfactant may not be sufficient to guarantee the dispersion of powders, but high values could reduce powder effectiveness because graphite is removed from the machining area. Also in this case, the ratio between graphite and surfactant equal to 0.3 gives the best result.

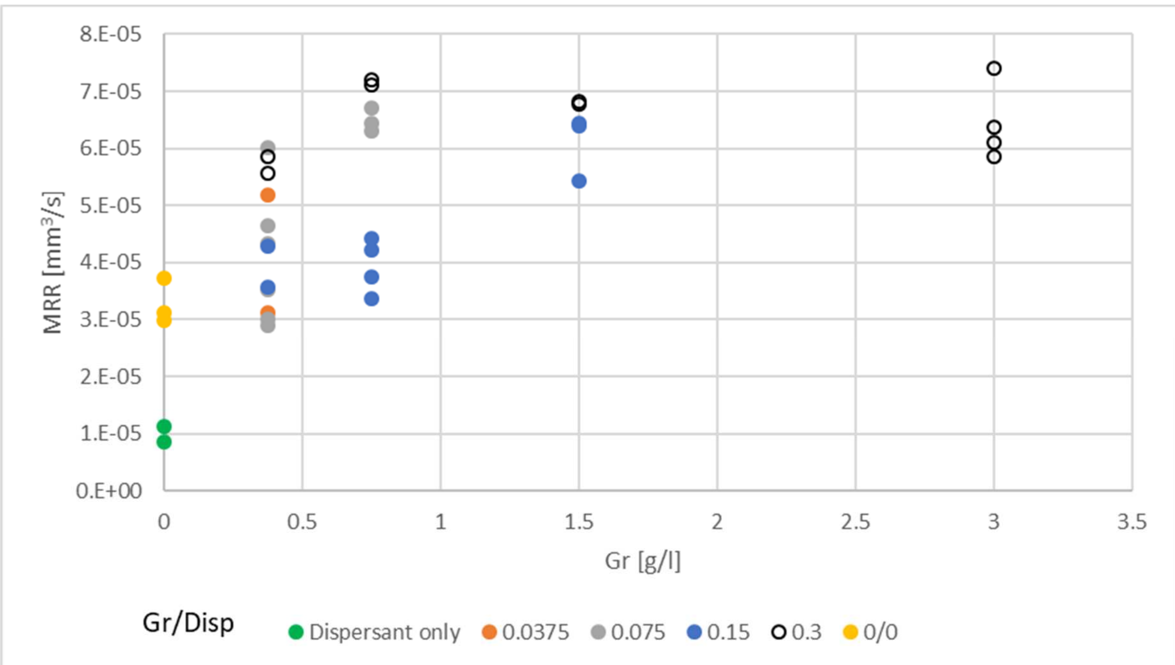

Fig. 4 MRR as a function of the graphite concentration varying the ratio between graphite and surfactant

Graphite also affects TWR (Fig. 5). Increasing its concentration, TWR decreases reaching values significantly lower than the reference case (around 1/3). Probably some chemical composts are formed during the sparks. The substances could migrate on the electrode surface, acting as a protective barrier reducing the electrode wear. The effect of the ratio between graphite and surfactant concentration is not evident in this case. Similar behaviour is also observed with 0% graphite concentration, showing that the effect of surfactant on TWR is minimal.

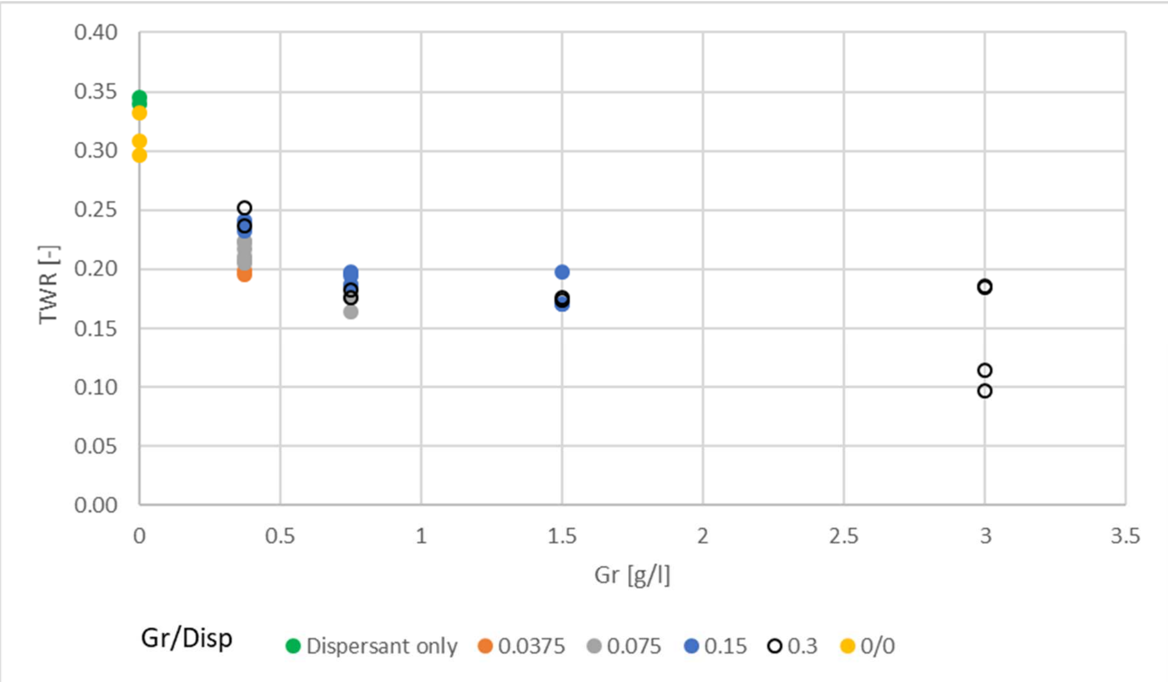

Fig. 5 TWR as a function of the graphite concentration varying the ratio between graphite and surfactant

As shown in Fig 6 and Fig 7, graphite in the dielectric influences the geometrical aspects of micro holes, causing larger top diameters than the reference case due to the higher frequency of sparks. This is confirmed considering the case of only surfactant (green points) with the lowest value of overcut. In fact, the graphite, released from the dissociation of kerosene during erosion, is dispersed quickly, slowing down the machining progress but guarantying a better accuracy. The graphite concentration seems not to have important effects on DOC. When graphite is added, the data scatter is higher than the reference case.

The taper rate gets worse when graphite is used except only in one case (1.5 g/l of graphite and the ratio of graphite and surfactant concentration equal to 0.3). Anyway, also for the taper rate, the ratio of 0.3 gives good results. It should be pointed out that the taper is also influenced by electrode wear modes (either radial or axial wear). Radial wear is a function of the axial coordinate and is likely to be affected by local graphite concentration (that may be variable along the electrode axis). In this paper, only axial wear was recorded since the direct measurement of electrode radial wear would require special techniques.

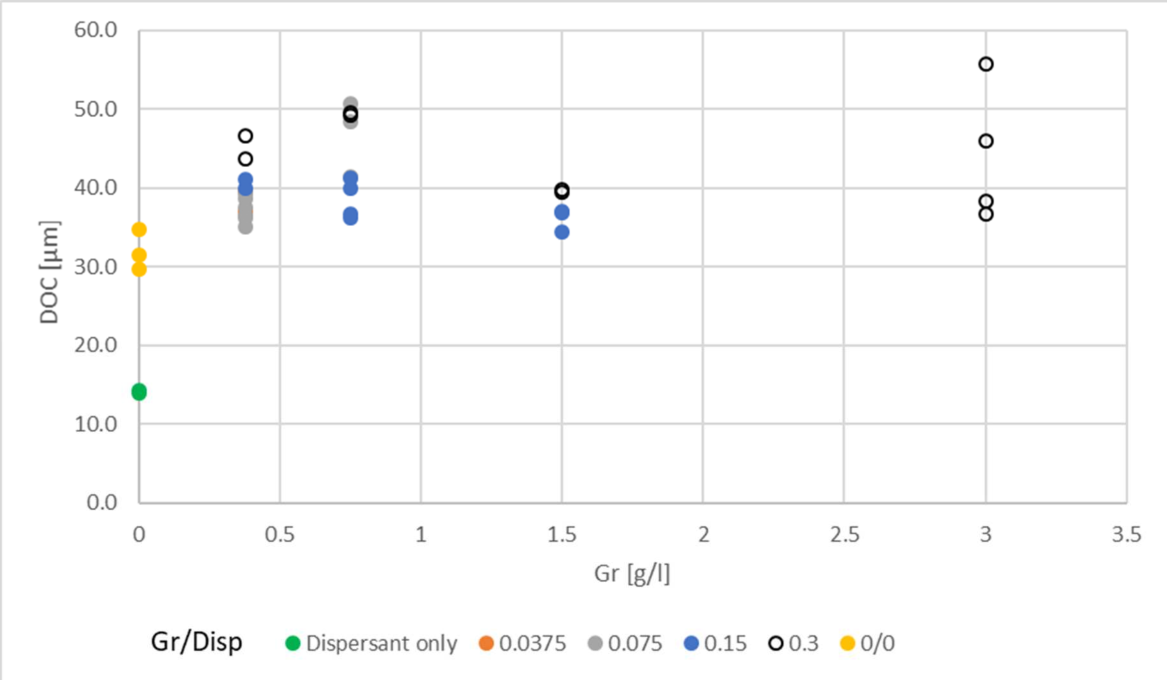

Fig. 6 DOC as a function of the graphite concentration varying the ratio between graphite and surfactant

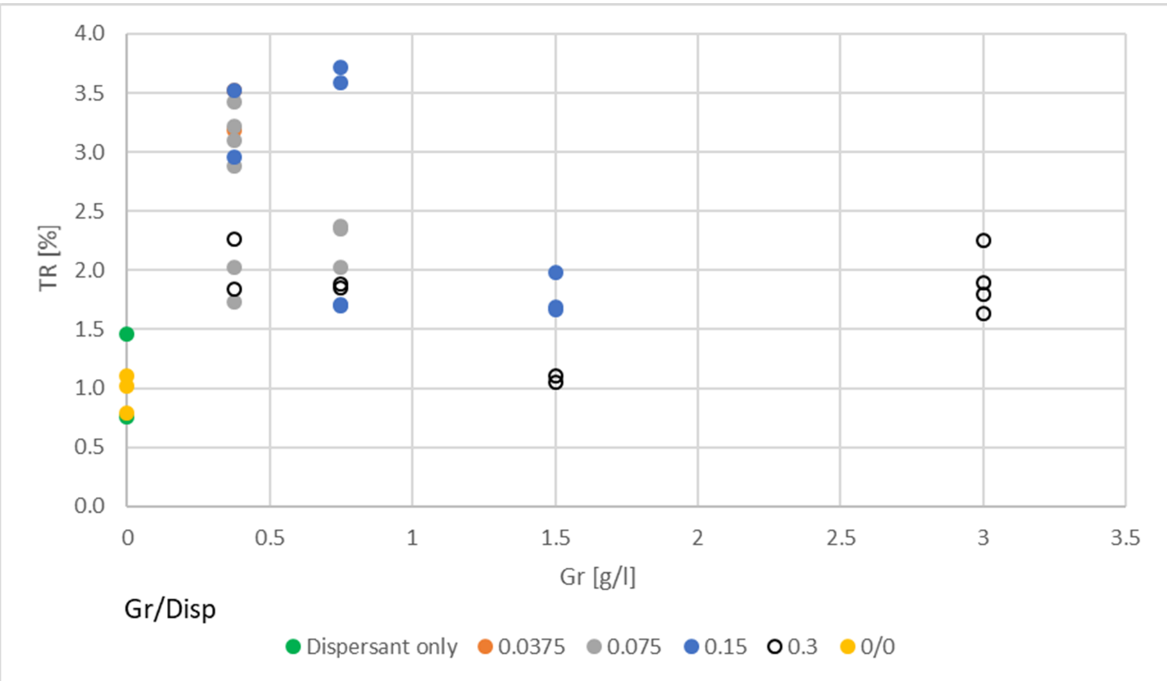

Fig. 7 TR as a function of the graphite concentration varying the ratio between graphite and surfactant

Conclusions

PMEDM is widely studied in the literature, considering the improvements in the process performance with respect to the traditional approaches. However, before extending this practice in the industrial field, solutions to guarantee better control of the process should be implemented. In this scenario, the effects of the concentrations of the additives in the dielectric fluid have to be better analysed. The paper aims to study the effects of powder and surfactant concentrations in the dielectric on the micro EDM drilling performance. Titanium sheets having a thickness of 0.5mm were drilled using a WC cylindrical electrode having a diameter of 0.1mm. To investigate the influence of their concentrations on the process, graphite and 4-dodecylbenzenesulfonic acid were used as powder and surfactant in the ranges between 0 – 3.5% and 0 – 10%, respectively.

Machining times are reduced by up to 30%, depending on graphite concentration (best results are found with Graphite concentration of both 1.5% and 3%). A wider data spread is observed at lower concentrations, especially when a higher amount of surfactant is tested. In such cases, the performances are less noticeable and sometimes even worse than with the base dielectric.

Electrode wear undergoes only minor changes. TWR, however, shows a remarkable trend, decreasing with graphite concentration. This behaviour is mainly due to the larger volume of holes drilled using graphite powder.

Hole sizes, evaluated through DOC, are indeed increased by adding graphite, but this effect is not proportional to the overall graphite content.

A special remark is due to the case in which only surfactant is added. As a result, machining time is increased by a large amount (more than twice that with the standard dielectric), tool wear is lower, and DOC is surprisingly small. All these behaviours may be explained by assuming that the surfactant removes the graphite produced by sparks in hydrocarbon dielectric, reducing its concentration to lower values than only hydrocarbon oil.

The effect of surfactant is also noticeable when the ratio of graphite/surfactant is changed. Best results are obtained using a ratio equal to 0.3. Decreasing such ratio, larger data scatter is always observed too. Possibly, not all graphite is available for the EDM process. Some graphite may be inhibited by the surfactant, whereas another fraction may be unavailable because of excessive aggregation. Adding surfactant increases inhibited graphite but decreases the aggregated particles as well. Such opposite effects of surfactant may contribute to explaining the observed data scatter. Values of graphite/surfactant ratio higher than 0.3 could not be tested because of graphite precipitation.

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Declarations
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The authors have no relevant financial or non-financial interests to disclose.

Author Contributions

All authors contributed to the study conception and design. Material preparation were performed by Giuseppe Rosace and Valentina Trovato. Experimental tests, data collection and analysis were performed by Giuseppe Pellegrini and Chiara Ravasio. The first draft of the manuscript was written by Giuseppe Pellegrini and Chiara Ravasio and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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