



Effect of Silane Treated Wheat Husk Biosilica (WHB) Deionized Water Dielectric on EDM Drilling of Ti-6Al-4 V Alloy

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Abstract

This present study deals about the effect of adding wheat husk biomass converted biosilica into the deionized dielectric medium and its machining effects on high performance engineering alloy Ti-6Al-4 V in electro discharge machining process. The biosilica particles were prepared from wheat husk via thermo-chemical method followed using silane treatment. The activated dielectric was prepared via shear stirring by adding considerable amount (0.25, 0.5 and 1.0wt.%) of biosilica particles. The machining characteristics such as material removal rate (MRR), tool wear rate (TWR), surface roughness (Ra) and microstructure were investigated. According to the results the silane treated biosilica of 1.0wt.% in deionized water gives improved MRR with controlled TWR and surface roughness whereas the as-received biosilica in deionized water produced lesser MRR with high surface roughness and tool wear. However beyond 1.0wt.% of biosilica in water may produce undesirable effects on the machining characteristics. The SEM microscopic images revealed smoothly machined surfaces with no heat affected zones for silane treated biosilica-water dielectric. These machining characteristics improved powder activated dielectric fluids are highly recommended when EDM process of hard alloys and plain metals.

Keywords EDM · Biosilica · Dielectric · MRR · TWR and surface roughness

1 Introduction

Electrical discharge machining (EDM) is a powerful, nontraditional machining technology that can machine any conductive material, regardless of mechanical properties [1]. EDM is a thermoelectric technique that uses a sequence of isolated sparks between the metal and the work piece to remove metal. The cutting tool in EDM is an electric spark, which is used to

cut (erode) the work piece and produce the completed item to the desired shape [2]. Though the EDM is powerful there are some notable drawbacks such as slow process, messy environment, pollution, poor surface finish, residual stress, and a heat-affected zone [3]. Researchers have proposed number of ways to improve EDM capabilities and to reduce the pollution by adopting new materials and technologies such as green dielectrics. Powder-mixed EDM (PMEDM) is a type of EDM that uses appropriate powder particles that are doped in a dielectric fluid while machining and acts as super coolers. The presence of powder could increase the spark length by effective bridging between the tool and work piece. Thus the MRR could be higher with more concentrated sparks with increased standoff distance. Since the standoff distance increases, the chances of reverse etching via the removed material between the tool and work piece also decreases. The large standoff distance presence at the gap of tool and work piece the trapped material could flush off effectively with higher dielectric flow. Hence the tool's life could be significantly increased. Moreover, using green dielectric one could reduce the amount of harmful wastes produced from the unconventional machining. Moreover, material machining, including superalloys and composites, are done effectively by these

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powder doped dielectric EDM techniques [4]. Joshi et al. [5] have done a feasibility analysis of powder-mixed deionized water as dielectric for machining Ti6Al4V. In this study alumina and SiC powder was doped into the deionized water. A response surface method is used to study the relationships between process parameters and response parameters. According to the results the peak current and pulse ON time is the most promising process parameter to achieve better MRR. Alam et al. [6] have done a performance evaluation of graphite and titanium oxide powder mixed dielectric for electric discharge machining of Ti-6Al-4 V. In this study the machining was performed using the copper electrode and kerosene as the dielectric medium with titanium oxide powder and graphene. Findings indicate that TiO₂ powder has a much higher effect on MRR compared to graphite powder. Similar study on experimental investigation of vegetable oil as dielectric fluid in electric discharge machining of Ti-6Al-4 V was done by Singaravel et al. [7]. The vegetable oil based dielectric fluids are considered namely, sunflower, canola, jatropha oils and conventional dielectric fluid namely kerosene are used in this investigation. In comparison to traditional dielectrics, vegetable oil possesses similar dielectric properties and erosion mechanisms, indicating that it might be used as a dielectric fluid in the EDM process. Moreover, the effect of powder mixed dielectric on material removal and surface modification in microelectric discharge machining of Ti-6Al-4 V was investigated by Kuriachen et al. [8]. In this the experiments were prepared and carried out using a response surface methodology (RSM)-Box-Behnken statistical design, with analysis of variance used to determine the importance of input parameters. According to the findings, a powder concentration of 5 g/L, a capacitance of 0.1 F, and a voltage of 115 V should be used to achieve maximum material removal and low tool wear. Similarly, Ishfaq et al. [9] have done a comprehensive analysis of the effect of graphene-based dielectric for sustainable electric discharge machining of Ti-6Al-4 V. The experiments with three types of electrodes such as aluminum, brass, and copper, were carried out using Taguchi's design (L18). Totally 36 trials were done out of which 18 using graphene-mixed dielectric and the rest using kerosene. The brass electrode with negative tool polarity produces larger MRR for both types of dielectrics, according to the results of the experiments. In comparison to kerosene (4.621 mm³/min), the greatest MRR attained with graphene mixed dielectric (7.602 mm³/min) is 64.5 % higher. Furthermore, the minimal TWR for graphene-based dielectric, 0.17 mg/min, is almost 1.5 times lower than for kerosene. Thus it is clear that the addition of nanoparticle into the water or kerosene as dopant improved the machining characteristics.

However using synthetic nanopowder for the purpose of enhancing the EDM process may create pollution to the environment and also poor for human health [10]. To overcome this researcher started using bio-derived nanoparticle for new engineering applications such as conventional machining and micro machining. These particles are eco-friendly, cost effective, sustainable, easy processing routes than the synthetic one [11]. Though the extraction routes of these bio particles are energy consuming their outcomes are more desirable for human beings. Moreover when mass production is implies the cost could be come down. Biochar, biosilica, green nano-silver, sea shell powder and egg shell powders are the notable fillers [12]. There are many routes to prepare these bio particles starting from domestic waste to agriculture biomass. Rice straw, rice husk, wheat husk, maize straw, sugarcane bagasse and domestic wastes are usually turns to the reactive functional materials via pyrolysis or thermo-chemical method [13]. Since these materials are abundant and making large landfill problems. Converting these biomasses as functional materials gain high process economy and solution to solid waste disposal issues [14]. Among various biomasses the wheat husk is a notable one. All over the world the demand for wheat and wheat based bi-products are high in demand, which eventually leave large volume of biomass. This biomass contains about 70 % of silica content and conversion of this may yield large silica volume [15]. This silica is thermally and chemically stable and reactive with organic solvents thus can be used in powder dielectric EDM applications [16]. However the high hydrophilic nature of silica would affects the machining characteristics. Thus the silica particles could surface treat before doping with dielectric medium. Aminosilanes are the one, which is functionally reactive and offers high grafting yield. This silane separates the particles against cluster and facilitates the particles functionally reactive (17, 18). Thus when these silane treated particles are doped into the deionized water there will not be any clusters and dielectric breakdown. Thus high quality arc could possible thereby high MRR and low TWR could be achieved. However in spite of many studies related to powder doped dielectric in EDM process the study using biosilica from biomass was not been researched by anyone. The effect of silane treated powder doped dielectric also not researched in EDM yet. Thus based on the previous research done and hypothesis the research gap for this present study was arrived. This study aims to investigate to hypothesis, firstly how the wheat husk biosilica doped dielectrics influences in the machining characteristics of EDM and secondly how the silane treated biosilica particles stand out from as-received one. These eco-friendly biomass converted particle activated EDM process may improve cleaner production, good ambience and higher process economy.

2 Experiment

2.1 Preparation of Silane Treated WHB

There are two processes to making wheat husk biosilica from biomass. To manufacture wheat husk ash, the procured wheat husk was first totally burned in a thermal reactor on a sand bed with a separate air supply unit at 700°C. The generated wheat husk ash was mixed with various normalities of NaOH solution at 80 °C and stirred continuously for 1 h to form sodium silicate solution in the second stage. At ambient temperature, the sodium silicate was titrated with 1 N HCL at a pH of 7. The silica gels were generated at the end of the stirring procedure and were allowed to age for 24 h. At the end of the operation, the aged silica gels were combined with distilled water to form silica slurry. To make xerogel silica, the slurries were rinsed many times in distilled water and soaked in a beaker for around 20 h at 70 degrees Celsius. After that, the xerogel silica was ground for many hours in a mortar to create fine size. A particle size analyzer was used to determine the size of the prepared biosilica particle, which was found to be 40–50nm [19].

The thermo-chemically produced wheat husk biosilica was then treated to an acid hydrolysis silane treatment process. 3-Aminopropyltrimethoxysilane (APTMS) with a molecular weight of 179.29 g/mol was acquired from Sigma Aldrich in the United States. The pH of the entire solution was adjusted in the range of 4.5 to 5.5 by mixing 95 % ethanol with 5 % water and acetic acid. To make an aqueous-silane solution, the required amount of silane (typically 2wt. %) was gently mixed into the aqueous solution drop by drop. The desired amount of biosilica particle was then added to the aqueous-silane solution and gently stirred for 10 min. The surplus aqueous-silane solution was manually decanted from the surface-treated biosilica particles, and the Si-O-Si structure was formed by drying them in a hot oven at 110 °C for 20 min [20]. The graphical illustration of silane treated wheat husk biosilica generated in this study is shown in Fig. 1.

2.2 Preparation of WHB-Water Dielectric

The dielectric fluid for this present study was prepared using silane surface treated wheat husk biosilica particles and deionized water. In this the biosilica particles are slowly mixed with deionized water and ultrasonically stirred for 10 min. The resulted solution was slightly heated up to 60°C and stirred for another 10 min to initiate the silane reaction with deionized water. Then the finely dispersed biosilica particles are cooled down to room temperature and ready for machining [21]. The designations of various dielectric fluid prepared in this present study are presented in Table 1. Figure 2 shows the molecular structure of silane treated biosilica and the reaction cite, where the water could possibly reacts to form a complete miscible admix.

2.3 Drilling Process

The micro EDM drilling was performed using an Ocean OCT-3525NA electrical discharge machine with prescribed drilling parameters described in Table 2. The machine has the capability of making micro holes of ranges from 0.3–0.5mm with 50 A pulse generator. The dielectric fluid used for drilling sample was deionized water, as-received biosilica with deionized water and silane-treated biosilica with deionized water respectively. The brass electrode of 0.47mm was used for making deep holes. The drilling process was done on Ti-6Al-4 V alloy of 10 mm thickness. Figure 3 shows the micro EDM machining setup utilized in this present investigation.

3 Characterization

The material removal rate of each experiment was calculated by taking before and after weight of material with respect to machining time. Similarly, the tool wear rate also calculated using mass loss of tool before and after drilling with respect to the time. The weight of both work piece and tool was weighed using a three decimal accurate digital balance machine. The machining time, which is used for calculating the MRR and TWR, was calculated using a digital stop watch. Moreover, the surface roughness of the machined surface was calculated using a surface roughness tester Mitutoyo version 2.0, JAPAN. The samples inner drilled portions were subjected for roughness measurement. The microstructure of machined surfaces was investigated using an scanning electron microscope, HITACHI, S1500, JAPAN with standard operating method.

4 Results & Discussion

4.1 Material Removal Rate (mm³/min)

Figure 4 shows the material removal rate of various forms of deionized biosilica activated dielectric fluid in machining of Ti-6Al-4 V alloy. It is noted that the pure water as dielectric produced significantly lesser material removal rate of 31.24mm³/mm. This lower material removal rate was the reason for super hard behavior of base metal and the less concentrated spark from tool towards the work piece. Thus lesser erosive force on the work piece couldn't make uniform material removal [22]. However the addition of wheat husk biosilica of as-received and silane treated form improved the MRR. A highest MRR of 41.8mm³/mm was observed machining process done using silane treated biosilica activated dielectric at specific machining condition. This is about 33.8 % of improvement on compare with the plain deionized water. Other dielectric fluid in EDM process gives improved MRR

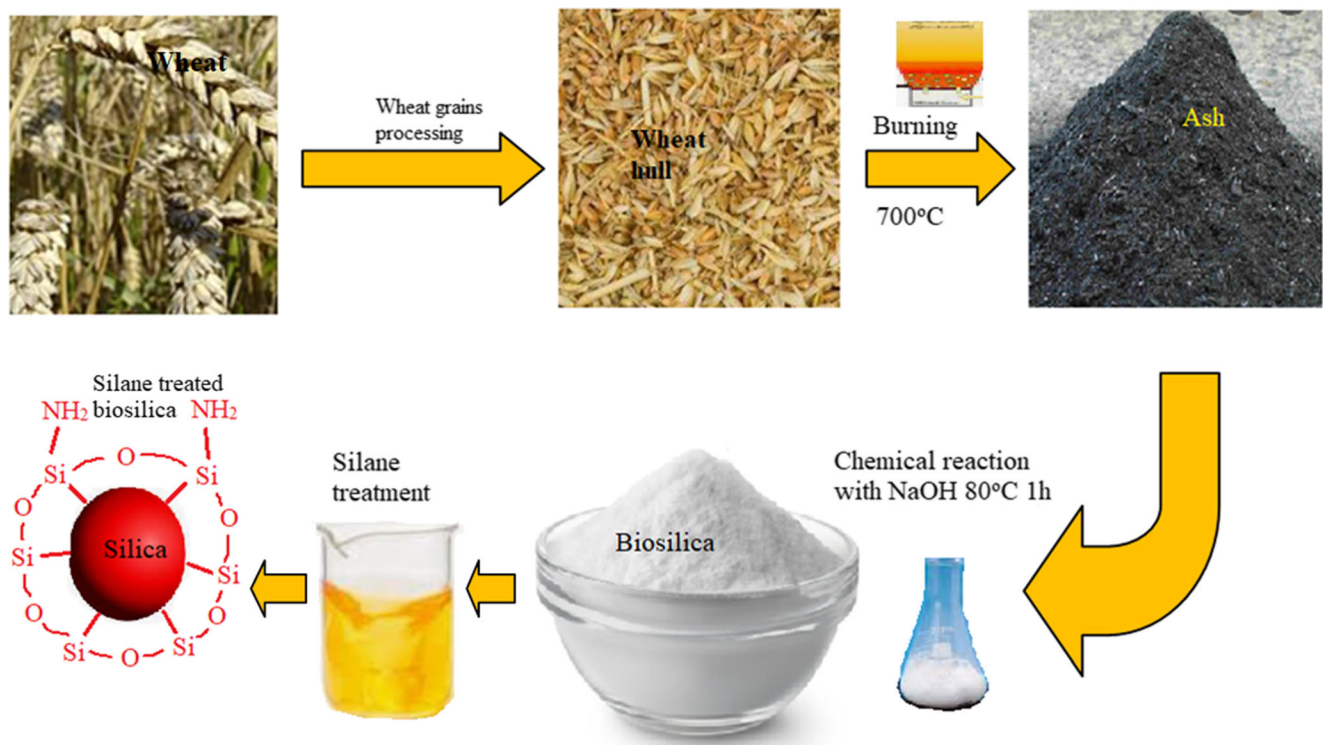


Fig. 1 Graphical illustration silane treated biosilica preparation

of 34.11, 37.35, 31.82, 36.38, 39.41 and 41.88mm³/min for D11, D12, D13, D21, D22 and D23 respectively. This is about 9.18 %, 19.55 %, 1.85 %, 16.45 %, 26.15 % and 34.05 % of improvement.

This notable improvement is the reason for effective spark length and higher standoff distance in powder dispersed dielectric fluid. When the biosilica particles are present between the tool tip and the work piece effective polarization takes place thus lengthier sparks used to produce [23]. Thus the amount of material removal from the material is higher. It is noted that in EDM process the silane treated biosilica particles enriched water dielectric produced higher MRR than as-received one. Even high volume of particle loaded up to 1.00 % there is significant MRR observed. But in as-received biosilica activated dielectric beyond 0.5wt.% the

MRR is decreased. Especially in nanofluid D13 (as-received), very lower MRR of 31.82mm³/min is observed. This lowest value is the reason for large dose of particle, which could agglomerate in aqueous medium and increase the erosion of tool material thereby affecting the MRR [24]. Similar effects were reported in the previous literature [24].

However in silane treated biosilica dispersed dielectric the prevention of particle agglomeration in the water dielectric increased the MRR. The silane treated particles uniformly arranged as polarity exist at the same time they never produce any continuous particle network whereas in as-received biosilica particle in deionized water more chances of particle agglomeration since the biosilica is highly hydrophilic. Thus

Table 1 Designations of water dielectrics

| Dielectric Designation | Biosilica form | Weight of biosilica (%) |
|------------------------|----------------|-------------------------|
| D ₀ | - | - |
| D ₁₁ | As-received | 0.25 |
| D ₁₂ | As-received | 0.50 |
| D ₁₃ | As-received | 1.00 |
| D ₂₁ | Silane-treated | 0.25 |
| D ₂₂ | Silane-treated | 0.50 |
| D ₂₃ | Silane-treated | 1.00 |

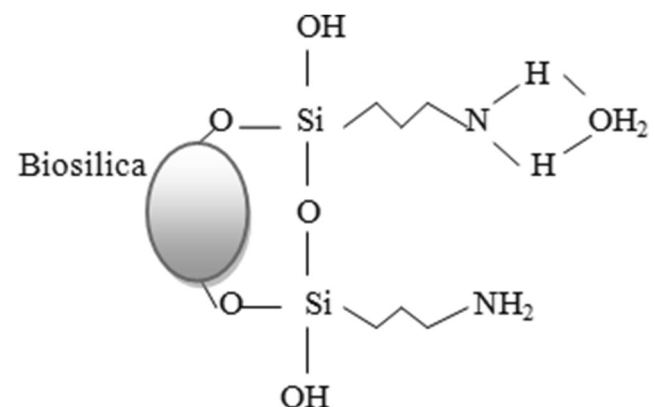
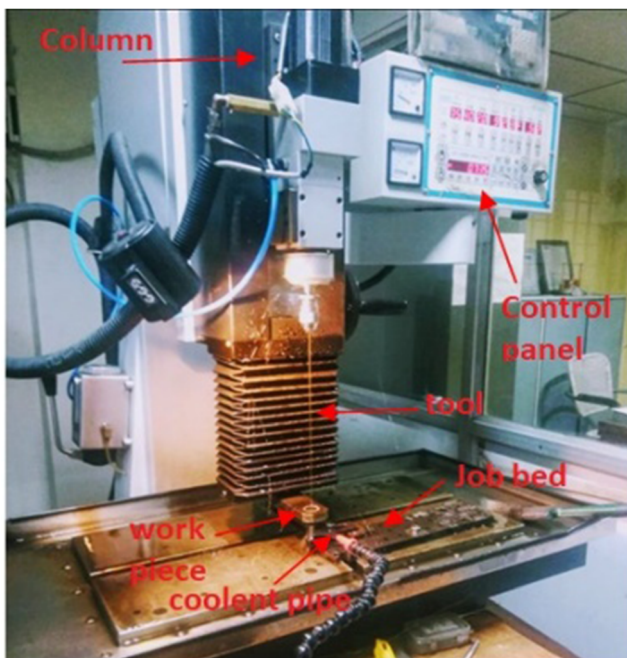
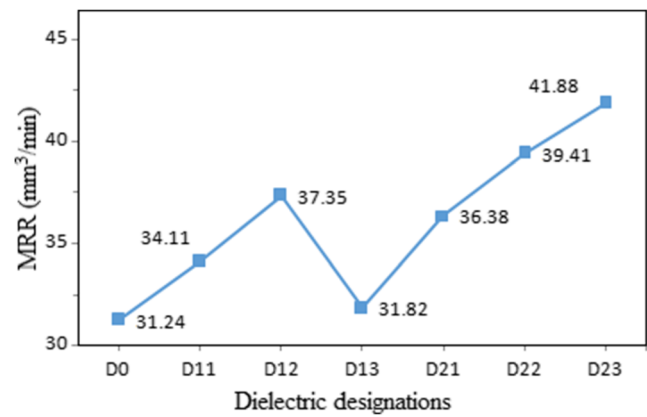


Fig. 2 Reaction of silane treated biosilica with water

Table 2 Description of process variables

| Process variable | Description |
|--------------------------|------------------|
| Pulse generator type | ISO energy pulse |
| Flushing pressure | 1 bar |
| Standoff distance | 8mm |
| Length of cut | 10mm |
| Gap voltage (V) | 50 |
| Capacitance (nF) | 6 |
| Pulse on time (μ s) | 50 |
| Duty factor | 0.6 |

higher agglomeration in the water medium enhances the biosilica particles settle in one place and restrict the uniform spark production and also the controlled length of it. Figure 3 shows the SEM microstructure of machined surfaces. Figure 5a shows machined surface of deionized water as dielectric. The surface is rough and high wavy with more dimples. This happens due to the unusual material removal from the work piece. However Fig. 5b and c shows the images of machined surfaces using biosilica dispersed water dielectric (D23). It is noted that in Fig. 3b the surface is very rough as plain dielectric, which confirms unusual material removal due to dielectric breakdown whereas in Fig. 3c the machined surface is smooth and ordered. There is no evidence for rapid material erosion due to uncontrolled sparks. This is due to the higher order of biosilica particles in the dielectric medium and continuously produced controlled sparks.

**Fig. 3** Micro EDM used in this present investigation**Fig. 4** MRR values in EDM drilling

4.2 Tool Wear Rate (mm³/min)

Figure 6 shows the tool wear rate of various dielectric fluid micro EDM process. It is noted that the tool wear rate of deionized water equipped EDM process gives considerably higher TWR of 0.336mm³/min. This is because of average heat absorption capability of deionized water. When the sparks are continuously produced the tool tip is subjected to thermal fatigue. If the tool is surrounded by high convective heat carrying dielectric fluid the rate of thermal fatigue could prevent thereby achieve less tool wear. The erosion wear mechanisms dominate the large tool material loss and provide improper machining behavior. However the presence of WHB particle of significant volume into the dielectric reduced the tool wear rate. However the as-received particles in the deionized water marginally decreased the TWR for smaller volume of particles and increased the TWR for larger volume of particles. It is noted that the 0.25wt.% of WHB into the dielectric gives reduced TWR of 0.302mm³/min. But the addition of 0.5 and 1.0wt.% of biosilica increased the wear rate further. This increment is because of highly agglomerated hydrophilic biosilica in the water medium. These agglomerated particles induce more sparks around the tool's tip thus eroded more tool material [24].

However the silane surface treated WHB into the water dielectric reduced the tool wear loss while machining. It is noted that the reduced TWR of 0.269, 0.215 and 0.242mm³/min was noted for D21, D22 and D23 dielectric fluid designations respectively. This is about 24.9 %, 56.27 % and 38.84 % of improvement in tool wear resistance. This improvement is the reason for effective polarization of biosilica particles between the tool's tip and the work piece [25]. Moreover being activated dielectric used in the machining process the tool is marginally off set from the work piece, which is quite larger than the usual. The biosilica fine particles in the dielectric absorb large heat and maintain the tool free from thermal fatigue. Thus the creation of unwanted

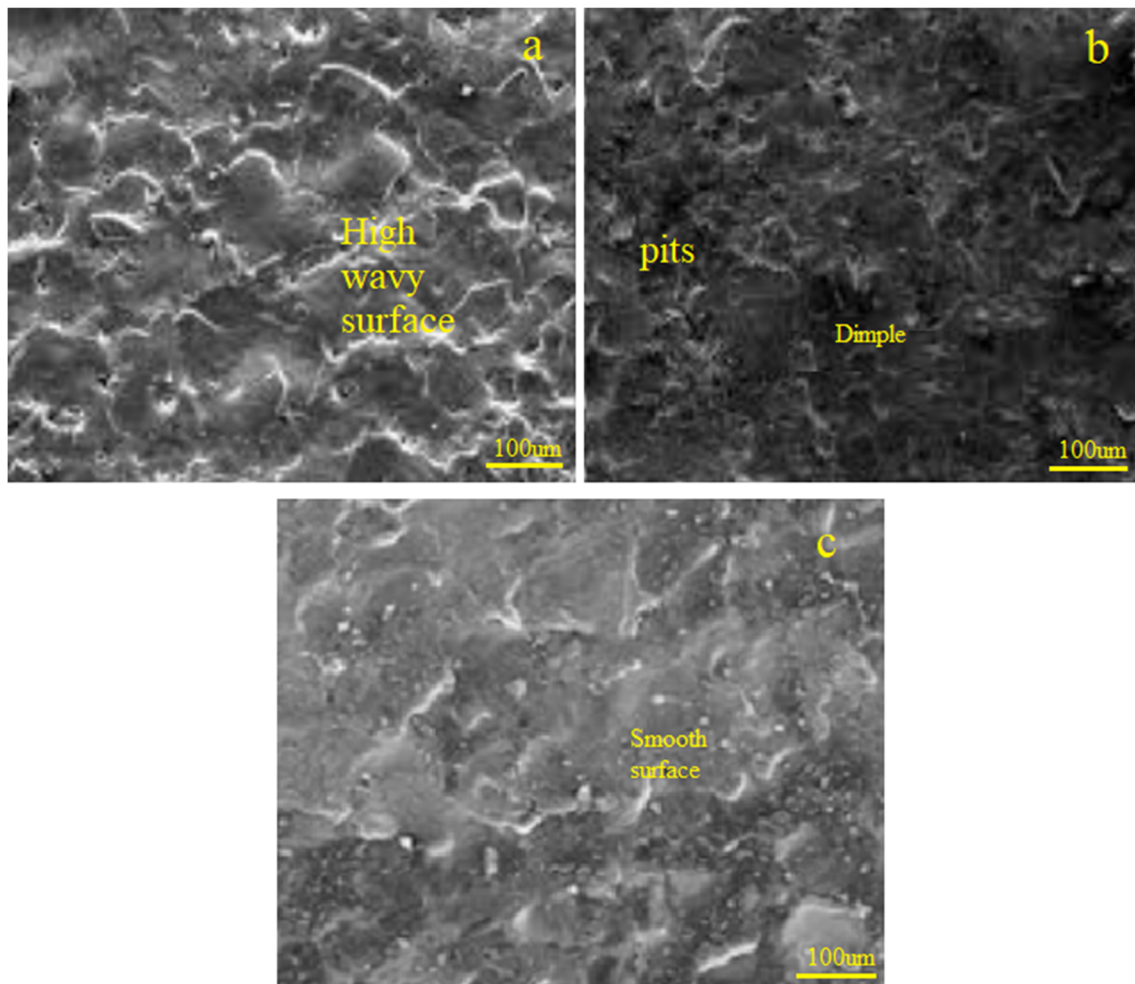


Fig. 5 SEM images of machined surfaces

secondary sparks and uncontrolled spark creations were hampered during the machining process, which facilitating highly stable tool's surface [26]. Even after long run the tool was not

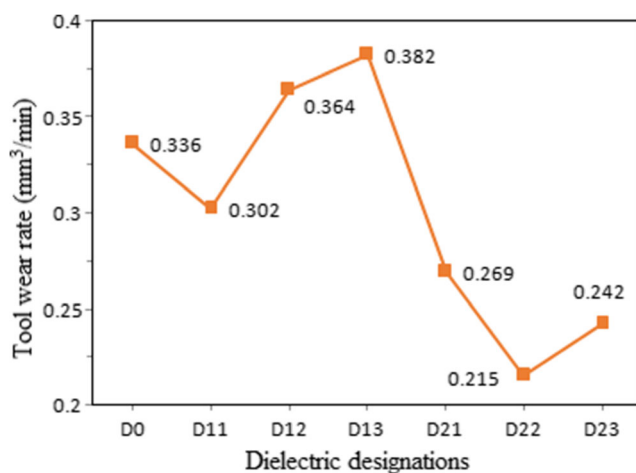


Fig. 6 TWR values in EDM drilling

eroded much and maintains its structural rigidity in the silane surface treated WHB added water dielectric. It is noted that both in D13 and D23 the reported TWR is marginally higher. Though the particles are silane surface treated the higher dose of biosilica particle in deionized water increased the degree of agglomeration. In that the non surface treated particle is agglomerated very high and leading to the reduction in MRR and increase in TWR. This notable agglomeration in the deionized water is the reason for the weak Van der waals attraction between the water particle and hydroxide state of biosilica [4]. Similar issues were reported in previous literature [4]. Author reported the agglomeration effect of biosilica particle in water based dielectric when machining the medical implants.

Figure 7 shows the SEM image of EDM tool before and after the machining. Figure 7(a) shows the unused brass tool. The tool's profile is neat and geometrically clear. But the EDM process performed using as-received WHB particle activated dielectric gives marginally poor structural metrics. The tool's edge is blunt and eroded away via thermal fatigue and

erosion wear loss (Fig. 7b). However the tool's edge at Fig. 7(c) shows high structural stability. The tool's surface is still retaining the actual dimensions as it was before. This is possibly the improved cooling effect, carry away of slurries and effective standoff distance.

4.3 Surface Roughness (μm)

Figure 8 shows the surface roughness values of various dielectric fluids employed EDM deep drilling process. It is noted that the plain deionized water as dielectric gives the higher surface roughness of $4.72 \mu\text{m}$. This higher surface roughness was the reason of improper evacuation of slurries formed during machining. These slurries again moved back to the material due to the inter-metallic attraction and damaged the machined surface. Moreover the less cooling effect of deionized water not able to maintain less cross pattern on the work piece thus offered high surface roughness.

It is noted that the presence of wheat husk biosilica particles of significant volume in the deionized water reduced the surface roughness. The decrement in surface finish of 40.89 %, 25.53 %, 8.75 %, 48.42 %, 111.6 % and 80.15 % were noted for EDM machining of titanium alloy using D11, D12, D13, D21, D22 and D23 respectively. In this The silane

treated WHB particles are seen giving lesser surface roughness than plain deionized dielectric and as-received biosilica dispersed water dielectric. This improvement in silane treated biosilica dispersed dielectric during deep drilling process was the reason of effective tool offset from the work piece. Thus the space between the tool and work piece good enough gap to evict all the removed material and reduces the chances to get collide each other [27]. This phenomenon eventually decreased the surface roughness of the machined surface and offered smooth surface. It is noted that the deionized water dielectric designation D13 shows highest surface roughness of $4.34 \mu\text{m}$ and it is almost closer to the plain deionized water. This highest surface roughness was the reason for higher particle agglomeration in dielectric. When large volume of particle is added in as-received form they tend to attract by each other and likely to form a bridged network. Thus the material removal was uncontrolled and witnessed higher cross pattern [28, 29]. However, the silane surface treated biosilica in the deionized water maintains high distinguish phase and controls the spark length and concentration. This eventually increased the surface smoothness.

Figure 9(a and b) shows the EDS graph of D12 and D22 dielectric activated machined surfaces of Ti-6Al-4 V alloy respectively. The peaks pertain with the molecules of only titanium, aluminum and vanadium. There is no evidence for

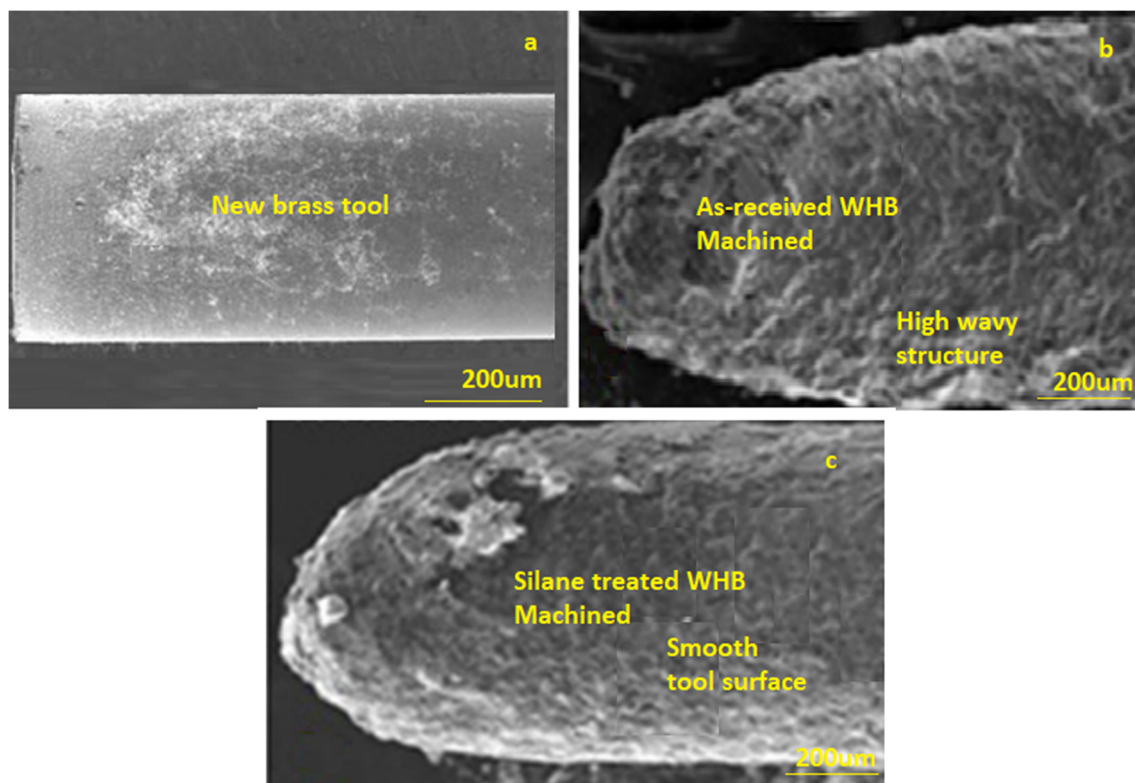


Fig. 7 SEM images of (a) as-received tool, (b) as-received WHB dielectric and (c) silane treated WHB dielectric machined

Fig. 8 surface roughness values in EDM process



silica particle impregnation during the machining process even though they are functionally active. Thus the WHB activated dielectric didn't alter any base properties of Ti-6Al-4 V alloy during machining.

5 Conclusions

This present study revealed the importance of adding silane surface treated wheat husk biosilica particles in deionized water dielectric at EDM deep hole making in Ti-6Al-4 V super hard alloy. The biosilica particles were silane surface treated using 3-Aminopropyltrimethoxysilane using acid hydrolysis method. The specific outcomes after the addition of biosilica particles in the deionized water are as follows. The addition of biosilica particles in the deionized water improved MRR. The addition of 1.0wt.% of silane surface treated biosilica gives highest MRR of 41.88mm³/min whereas the as-received biosilica in deionized water

dielectric gives lesser MRR. The silane treated biosilica activated dielectric in EDM process gives lesser tool wear rate of 0.215mm³/min for 0.5wt.% of silane treated WHB in water. However the as-received biosilica particles eroded the tool material larger. Similarly, the surface roughness of D22 dielectric enabled machined surface gives lesser surface roughness. Same amount of as-received biosilica in water gives higher surface roughness. The machined surface morphology revealed smooth, pit and dimple free surface for silane treated biosilica activated dielectric in EDM. Moreover the tool's surface morphology explicates the high structural integrity for silane treated D22 dielectric in EDM. The EDS spectra confirmed no silica impregnate into the Ti-6Al-4 V alloy base material. As the summary the biogenic route made silica nanoparticles from large biomass wheat husk acted as activator at deionized water dielectric in EDM process and amended the machining characteristics in positive way. Thus in the process of machining hard Ti-6Al-4 V alloys using unconventional machining process

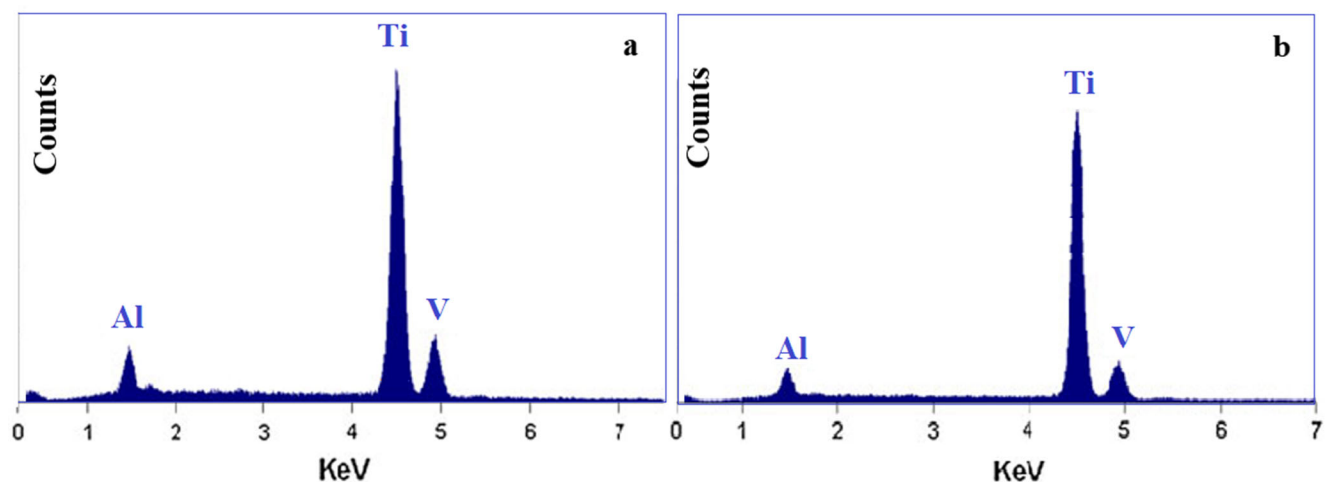


Fig. 9 EDS spectrum of machined surfaces

like EDM the powder dispersed water dielectric could be used to improve the machining characteristics.

Author Contributions All have done equal contribution.

Data Availability No data available to deposit as private. There are no rights.

Declarations Yes this article compliance with ethical standards of journal.

Conflicts of Interest/Competing Interests There is no conflict of interest by any form for this manuscript.

Consent to Participate Yes. All permission granted.

Consent for Publication Yes. All permission granted.

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