



## Characterization of polyethylene wear particle: The impact of methodology



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### ABSTRACT

Due to the prevalence of problems caused by wear particles, the reduced durability of total joint replacements is well documented. The characterization of wear debris enables the size and morphology of these wear particles to be measured and provides an assessment of the biological response in vivo. However, the impact of different methodologies of particle analysis is not yet clear. Hence, the aim of this investigation was to analyze the influence of different particle characterization methods performed by three research centers within the scope of a “round robin test”. To obtain knowledge about possible pitfalls, single steps of the particle characterization process (storage, pore size of the filter, coating durations by gold sputtering and scanning electron microscopy (SEM) magnification) were analyzed. The round robin test showed significant differences between the research groups, especially for the morphology of the particles. The SEM magnification was identified as having the greatest influence on the size and shape of the particles, followed by the storage conditions of the wear particle containing lubricant. Gold sputter coating and filter pore size also exhibit significant effects. However, even though they are statistically significant, it should be emphasized that the differences are small. In conclusion, particle characterization is a complex analytical method with a multiplicity of influencing factors. It becomes apparent that a comparison of wear particle results between different research groups is challenging.

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## 1. Introduction

Polyethylene (PE) as a bearing material for total joint replacement (TJR) has been the gold standard for the past 40 years. However, it has become apparent that PE wear and the biological responses to wear products are limiting factors for the longevity of TJRs [1,2]. For this reason research has focused on PE wear particle analysis. There is a wide range of results regarding the size and morphology of in vitro [3–12] and in vivo [3,13–30] generated wear particles. Due to the investigations of new bearing surface materials (e.g. cross-linked PE) and optimization of the manufacturing process (e.g. sterilization in an inert atmosphere), today PE generates lower wear rates and produces smaller wear particles [31].

In order to characterize the wear particles, a complex methodological approach is needed which apparently differs between different research groups (RGs). It is not known if and to what extent

the methodology used has an impact on the results of the particle characterization.

In the present study a round robin test of wear particle characterization according to the ISO 17853: 2011 [32] standard was performed by three tribological RGs. The aim was to compare the results generated by particle characterization and to examine pitfalls which might have an influence on the particle measurement process, specifically (i) the storage of the wear particle containing lubricant; (ii) the pore size of the filter membranes used; (iii) the influence of different gold sputter coatings; as well as (iv) the magnification used for the visualization of particles.

## 2. Materials and methods

### 2.1. Round robin test

For the present investigation, test lubricant (diluted newborn calf serum; protein content 25 g l<sup>-1</sup>) containing PE wear debris of a previous knee wear simulator test was used. The medium was ultrasonically mixed until the debris was homogeneously distributed in the fluid and then divided into three portions. The blinded

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samples were subsequently sent to three different tribological RGs with established methods for wear particle characterization from wear simulator tests [5,10,33–37]. Each center used its standardized method and hardware as summarized in Table 1.

The RGs digested the test medium as described by Niedzwiecki et al. [38]. The test medium was stirred and 37% hydrochloric acid was added to the test medium with a ratio of 1:5. The digestion of the proteins occurred under continuous stirring at 60 °C for ~60 min. Afterwards, the digested fluid was diluted with methanol at the ratio of minimum 1:10. The methanol containing the wear debris was vacuum filtered through a filter membrane. Before obtaining the scanning electron microscopy (SEM) images, two of three centers coated the filters with a thin gold sputtering layer. Images were recorded by a magnification of 10,000×. All centers used the same digital image analyzing software (QWin, Leica, Wetzlar, Germany) to measure the size and morphology of the particles. The equivalent circle diameter (ECD), aspect ratio (AR) and roundness (R) were specified according to ASTM F 1877-05 (2010) described by Eqs (1)–(3) [39]:

$$ECD = 2 \cdot \sqrt{\frac{Area_{particle}}{\pi}} \quad (1)$$

$$AR = \frac{maximal\ diameter_{particle}}{minimal\ diameter_{particle}} \quad (2)$$

$$R = \frac{Perimeter_{particle}^2}{4 \cdot \pi \cdot Area_{particle}} \quad (3)$$

## 2.2. Potential

### 2.2.1. Influence of storage conditions of the wear lubricant (RG B)

After completion of a wear test, the lubricant was stored after wear testing for different times and at different temperatures:

- No storage; serum was analyzed directly out of the simulator.
- Storage of the serum for 6 months at room temperature (RT).
- Storage of the serum for 6 months at –20 °C.

The digestion of the lubricant, filtration and characterization of the wear particles were done as described above (Section 2.1).

### 2.2.2. Influence of the filter pore size (RG A)

Wear particles were isolated from the lubricant and filtered through an aluminum oxide filter membrane (Whatman, Anodisc, Maidstone, UK) with two different pore sizes: 100 nm and 20 nm. Afterwards, particles were characterized as described in Section 2.1.

**Table 1**

Technical and analytical specifications of the three research groups (RGs).

	RG A	RG B	RG C
Storage	Approx. 12 months at room temperature		
Digestion	Acid digestion based on Niedzwiecki et al. [38]		
Filtration	20 nm (Whatman, Anodisc, Maidstone, UK)	50 nm (Pieper Filter GmbH, Bad Zwischenahn, Germany)	20 nm (Whatman, Anodisc, Maidstone, UK)
Coating/ sputtering	No	Yes (gold)	Yes (gold)
Visualization	FEG SEM (LEO 1530, Carl Zeiss, Oberkochen, Germany)	SEM (DSM 960A, Carl Zeiss, Oberkochen, Germany)	SEM (EVO LS 10, Carl Zeiss, Oberkochen, Germany)
Magnification	10,000×		
Post-processing	QWin (Leica, Wetzlar, Germany)		

### 2.2.3. Influence of the coating by gold sputtering (RG C)

To measure the influence of gold sputtering, standard polystyrol particles (BS-Partikel, Wiesbaden, Germany) with a diameter of  $0.246 \pm 0.006 \mu\text{m}$  suspended in water were filtered and analyzed with three different sputtering durations, 1.5, 3.0 and 4.5 min, using a sputter coater (SCD 050, Bal-Tec, Scotia, USA).

The ECD was measured with the digital image analyzing software described in Section 2.1.

Additionally, to measure the thickness of the gold layer, silicon wafers were masked on one side with a tape and coated by gold sputtering in the prior described durations. The tape was carefully removed and the silicon wafers were analyzed by atomic force microscopy (AFM; JPK Instruments, Berlin, Germany).

### 2.2.4. Influence of the magnification by SEM (RG A)

Wear particle containing lubricant was processed as described above (Section 2.1). To investigate the influence of the SEM magnification used the lubricant was filtered through a 20 nm filter. Wear particle analyses were carried out using three different SEM magnifications: 5000× (17 pxl  $\mu\text{m}^{-1}$ ); 15,000× (50 pxl  $\mu\text{m}^{-1}$ ) and 30,000× (101 pxl  $\mu\text{m}^{-1}$ ).

## 2.3. Statistical analyses

The size and shape of the particles were measured using a grey-scale detection method with digital image analysis software (QWin, Leica, Wetzlar, Germany). All values were presented as mean value  $\pm$  standard deviation.

For two groups a non-dependent *t*-test (SPSS, IBM, Armonk, USA) was carried out to detect significant differences of the means. Furthermore, an ANOVA was used to compare mean values for more than two groups. For multiple-group comparisons a Bonferroni post hoc test was performed. *P*-values of <0.05 were considered significant.

## 3. Results

### 3.1. Round robin test

Fig. 1 shows typical SEM images of polyethylene particles of the three research groups (RG A, RG B, RG C). Additionally, the measured particle characteristics of the different RGs are shown in Table 2.

The measured mean particle ECD of RG A was comparable to RG B ( $P = 0.16$ ) and RG C ( $P = 0.15$ ). However, the mean ECD differed significantly ( $P = 0.02$ ) between RG B and RG C. Roundness and aspect ratio were significantly different between all RGs with  $P < 0.001$ .

In the histogram (Fig. 2) the frequency of the ECD is shown. All RGs measured a single mode particle distribution with a mode of 0.1  $\mu\text{m}$ . On the one hand RG A found particles with an ECD smaller than 0.03  $\mu\text{m}$ ; on the other hand RG B and C found particles larger

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