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# Tribological Properties of Poly(vinyl alcohol) Hydrogel in Response to Ceramic Femoral Component

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## ABSTRACT

Many biomaterials have been proposed as artificial articular cartilage in partial or total joint replacement. Poly(vinyl alcohol) (PVA) hydrogel is a kind of artificial cartilage and it exhibits excellent mechanical and biocompatibility properties. In this study, the friction tests under reciprocating motion were conducted to determine the tribological properties of PVA hydrogel in response to ceramic femoral component. The friction coefficient testing of PVA hydrogel against ceramic femoral component was performed by a three-factor, two-level designed experiment. The three factors studied were load, lubrication and sliding speed. Longer term friction tests were conducted under constant load and cyclic load to evaluate friction variations in 30 min. The friction coefficient between PVA hydrogel and ceramic femoral component was found to depend significantly on load, lubrication and sliding speed. The friction coefficient increased as load and sliding speed increased. The friction coefficient decreased when the lubricant was changed from Ringer's solution (RS) to hyaluronic acid (HA). In longer term tests, the friction coefficient in constant load tests was significantly lower that in cyclic load tests. The wear of PVA hydrogel under constant load was identified by environmental scanning electron microscopy (ESEM). It is indicated that the biphasic lubrication property is the key feature for PVA hydrogel. These results may be useful in the tribological design of artificial cartilage in joint replacement.

**Key Words:**

hydrogels;
biomaterials;
tribology;
lubrication;
artificial cartilage.

## INTRODUCTION

Articular cartilage has excellent friction and lubricating properties. Friction coefficient of articular cartilage is as low as 0.005 to 0.023 [1]. Engineers have done many research works on articular cartilage, but they have not been able to replicate its low friction in any man-made bearing. The damages of articular cartilage are common in trauma and arthritis. Thousands of people suffer pain caused by arthritis each year [2]. Arthritis has more than 100 different types, such as osteoarthritis (OA) and rheumatoid arthritis. In the United States, arthritis is the most common cause for disability, which affects 70 million adults. It is estimated that 90% of the human population suffer OA by the age of 70 years [3]. With arthritis, the damage or loss of articular cartilage alters its lubricating function. As a result, patients feel pain and uncomfortable in their

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everyday life.

Many kinds of synthetic articular cartilage, such as methyl methacrylate, poly(2-hydroxyethyl) methacrylate (polyHEMA) hydrogel, poly(vinyl alcohol) (PVA) hydrogel, etc., are developed to repair or replace the damaged articular cartilage [4-7]. PVA hydrogel, which has a three-dimensional network structure and large amounts of water without dissolution, has been widely used in medicine for biomaterials [8-10]. PVA hydrogel has many extraordinary properties, such as good biocompatibility, low elastic modulus and high resiliency. Because of these excellent properties, PVA hydrogel has been widely used to repair some kinds of tissues including articular cartilage. In some studies, PVA hydrogel has been prepared by the freezing-thawing process of PVA water solution [11,12]. PVA hydrogel was also prepared by a freezing process using an aqueous solution of dimethyl sulphoxide (DMSO) and PVA. Trieu et al. [13] have reported several variations in the preparation of PVA hydrogel. They found that PVA hydrogel prepared without DMSO has porous structure with local orientation, which was due to phase separation during the quenching period. In the solutions of PVA with DMSO, freezing depression occurred. Freezing depression allowed PVA solution to form a gel without experiencing phase separation. High cooling rate and rapid gelation occurs due to quenching at low temperature. PVA hydrogel prepared by DMSO solution shows a more homogeneous structure than a PVA hydrogel prepared without DMSO.

In joint replacement, one type of failure mode is osteolysis which is caused by wear debris and leads to loosening and instability of components [14]. In order to minimize the wear in components, ceramic femoral components are used as replacements. Ceramic femoral components have many excellent properties, such as hardness, high lubrication, wettability and biocompatibility. Although ceramic femoral components have been used for several years, the frictional property between ceramic femoral component and artificial articular cartilage is not clear. Moreover, the loads on natural synovial joints under motion are cyclical. It is necessary to study the long term friction properties of artificial cartilage under cyclic load.

The primary goal of this investigation was to determine the tribological properties of PVA hydrogel in response to ceramic femoral component under various experimental conditions. Friction coefficient testing of PVA hydrogel against ceramic femoral component was performed by a three-factor, two-level designed experiment to determine the effects of applied normal load, lubrication and sliding speed. Longer term friction tests under constant load and cyclic load were conducted upon PVA hydrogel-on-ceramic contact to evaluate the friction variations in a long time. The changes in the specimen surface were also observed.

## EXPERIMENTAL

### Materials

PVA and DMSO were supplied by Sinopharm Chemical Reagent Co., Ltd. (SCRC), PR China. The viscosity-average degree of polymerization of PVA purchased was 1750±50. The degree of saponification of PVA was 99%. The ceramic femoral component used in this research was 32 mm in diameter made of alumina (BIOLOX® forte, CeramTec AG, Germany). The ceramic femoral component had a highly polished surface with average roughness < 0.02 µm. Hyaluronic acid (HA) with the molecular weight of $2\times10^6$ Da was a commercially available sodium hyaluronate of Charoen Pokphand Group, Thailand. Ringer's solution (RS) was supplied by Tianrui Pharmaceutcal Corporation, Ltd., PR China.

### Methods

*Preparation of Specimen*

The solution of PVA was prepared by dissolving PVA in 25 wt% solution of DMSO in water [13,15]. The concentration of PVA powder was 15 wt%. First, the mixed solvent was stirred continuously at 110ºC for 3 h until the mixed solvent became a viscous slurry. Then the slurry was poured into stainless steel container and put in a medical refrigerator for 24 h at -20ºC. Following the freezing step, the PVA hydrogel was obtained by thawing the viscous slurry. Then the PVA hydrogel was immersed in distilled water which was changed continuously at 20ºC for 20 days to exchange the mixed solvent in the specimens. PVA hydrogel was placed in distilled water before tests. The dimensions of the specimens were 4 mm in thickness, 20 mm in width and 40 mm in length.

*Tribological Testing*

The friction experiments were performed at room temperature. The test machine was UMT-2 Micro-Tribometer (CETR Inc., USA). Friction force was measured by force sensors and converted into friction coefficient. Special stainless steel chamber and fixtures were designed to fix the sample. The ceramic femoral component was held by a conical fixture which was similar to artificial joint installation. PVA hydrogel samples were held by stainless steel fixture without any movement in tests. During these tests, the samples were kept immersing in the lubricant which was retained in the stainless steel chamber. The ceramic femoral component was fixed to reciprocate over a distance of 8 mm. A simplified sketch of the device is shown in Figure 1.

A three-factor, two-level designed experiment was conducted to determine the influence of different factors on the friction between PVA hydrogel and ceramic femoral component. The three factors and values of the two levels are summarized in Table 1. In this designed experiment, eight experimental configurations were necessary to complete the study. PVA hydrogel and ceramic femoral component maintained contact at 5 s before all the friction tests and the test time was 5 min.

In this study, HA was investigated as lubricant to compare with RS. One of the treatments for early stage OA is the injections of HA. The American College of Rheumatology has recommended HA for the medical management of OA of the hip and knee [16]. HA is an integral part of synovial fluid. HA was dissolved in physiological saline at a concentration of 4.0 mg/mL to simulate synovial fluid [17]. Hertzian contact assumptions were used in pressure of theoretical calculation. Loads were tested at 5 N and 10 N, which correlated to pressures of approximately 0.94-1.19 MPa. Loads lied in the normal range of stresses in human joints (0-5 MPa) [18]. The sliding speeds were 1 mm/s and 4 mm/s which were adopted in this test to ensure the contacts maintained within a mixed or boundary lubrication regime [19].

**Table 1.** Factors and levels for designed experiment.

| Factor | Low level | High level |
|---|---|---|
| Load (N) | 5 | 10 |
| Speed (mm/s) | 1 | 4 |
| Lubrication | Ringer's solution | Hyaluronic acid (4.0 mg/mL) |

In longer term friction tests, two kinds of load were used in these tests, including constant load and cyclic load. In constant load tests, the load was 10 N and sliding speed was 4 mm/s with RS or HA lubricated. In the cyclic load tests, a new experimental method was used and the friction pair was lubricated by HA. A complete cycle in the tests is shown below. First, constant load of 10 N was applied to the friction pair for 1 s without moving. Second, the ceramic femoral component moved 8 mm forward at 4 mm/s. Third, the ceramic femoral component moved back 8 mm at 4 mm/s. Finally, the ceramic

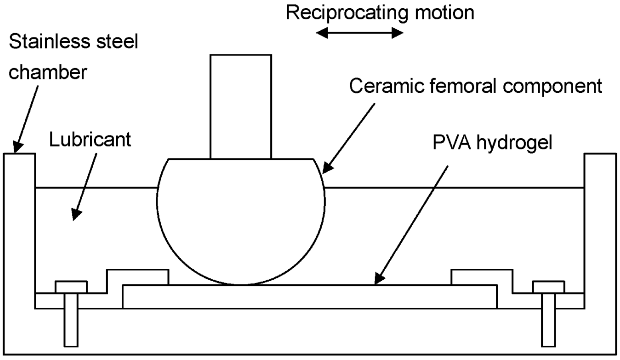


**Figure 1.** Device for friction testing.

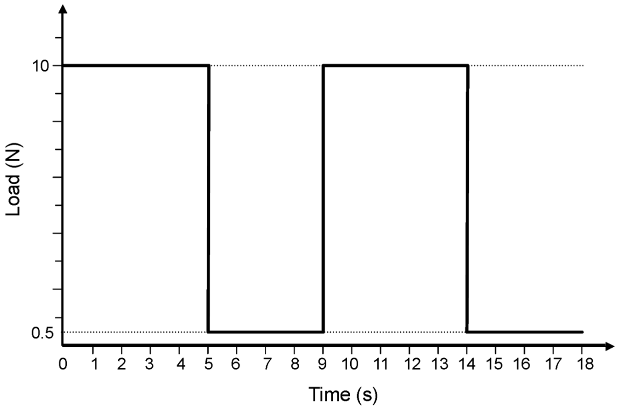


**Figure 2.** Schematic illustration of cyclic load.

femoral component stopped moving and the load was reduced to 0.5 N for 4 s. In Figure 2, a schematic illustration of the cyclic load in two cycles is shown.

### *Measurement*

Dynamic frictional properties were investigated for each of these tests. Friction data were obtained from the last 5 s of the data acquisition period. The friction coefficient was calculated from u = F/N, where u, F and N represent friction coefficient, friction force and applied load, respectively. The friction coefficient was the average value of the tests of each experimental configuration. Three specimens were tested within each combination.

### *Surface Analysis*

In order to evaluate the surface morphology changes in longer term tests, the specimens under constant load were examined by electron microscopy. In conventional scanning electron microscopy (SEM), lengthy preparations were required, including fixing, dehydration, drying and gold coating. It is inconvenient to examine PVA hydrogel by SEM due to its high water content. No special preparation was necessary for the examination in environmental scanning electron microscopy (ESEM). In this study, ESEM (Philips XL30) was used for the surface examination. The accelerating voltage, the spot size and the chamber pressure were 20 kV, 4.0-5.0 and 0.6-1.0 Torr, respectively. The temperature was maintained at room temperature. Surface roughness of specimens was also measured by TR100 Profilometer (TIME Group Inc., PR China). Scanning length of surface roughness was 6 mm, and sampling length was 0.8 mm. The profilometer produced no detectable surface damage to the sample surface, good repeatability, and allowed to measure within a short time to avoid dehydration. All specimens were measured before and after tests.

## RESULTS AND DISCUSSION

### The Three-factor, Two-level Designed Experiment

#### *Average Value of Friction Coefficient at Various Levels*

The influence of different factors on the friction coefficient of PVA hydrogel against ceramic is shown by the bar graph in Figure 3. The bar means the average value of the friction coefficient at various levels of different factors. The three factors tested have different influence on the friction. It is shown that the friction coefficient increased with load and speed, but decreased as lubrication changed. The average friction coefficient was 0.046 at low level load and 0.061 at high level load. The friction coefficient was 0.062 under RS lubrication and 0.045 under HA lubrication. The average friction coefficient at high level sliding speed was 0.066, which was almost 1.5 times of that at low level sliding speed.

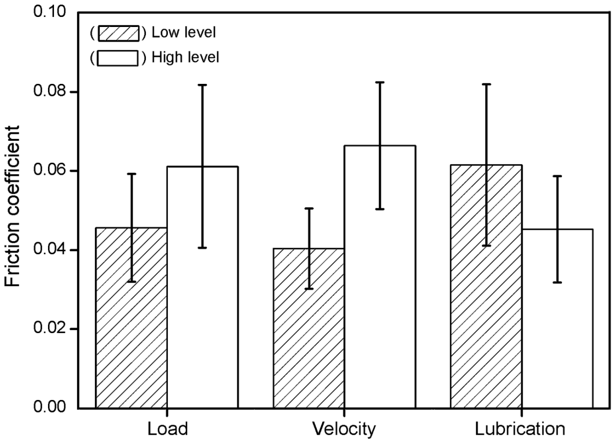


**Figure 3.** Main effects of the factors for the friction coefficient.

### *Effect of Load*

The bar graph in Figure 4 shows the friction coefficient of PVA hydrogel against ceramic femoral component with various loads at 5 N and 10 N, respectively. When load increased, the friction coefficient significantly increased. It can be observed that as load increased from 5 N to 10 N with the speed of 4 mm/s, the friction coefficient increased from 0.063 to 0.09 at RS lubrication, or from 0.051 to 0.062 at HA lubrication.

### *Effect of Sliding Speed*

General trends in the friction coefficient at different levels of sliding speed are illustrated by the bar graph in Figure 5. When the sliding speed increased, the increase of the friction coefficient was proportional to

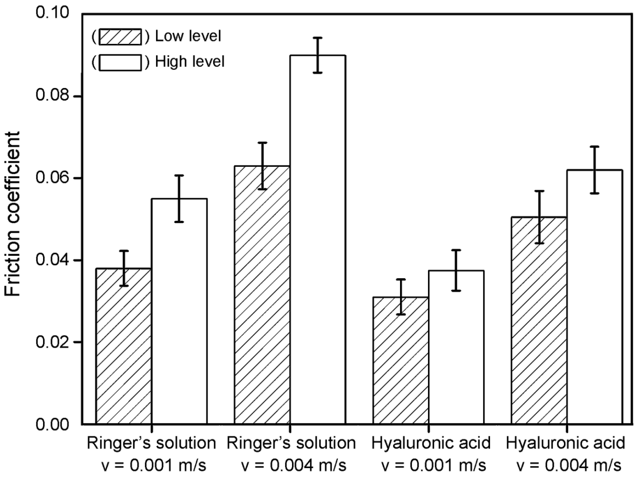


**Figure 4.** Effect of load on the friction coefficient; v is the sliding speed of each test.

the speed increase. It is shown that as sliding speed of ceramic femoral component increased from 1 mm/s to 4 mm/s, the friction coefficient of PVA hydrogel increased from 0.055 to 0.09 with RS as the lubricant.

*Effect of Lubrication*

Figure 6 shows the change of the friction coefficient with different lubricants. This graph shows that the effects of lubrication were obvious. It can be observed that as lubricant changed from RS to HA with the speed of 4 mm/s, the friction coefficient decreased from 0.063 to 0.051 at 5 N loads, or from 0.09 to 0.062 at 10 N loads.

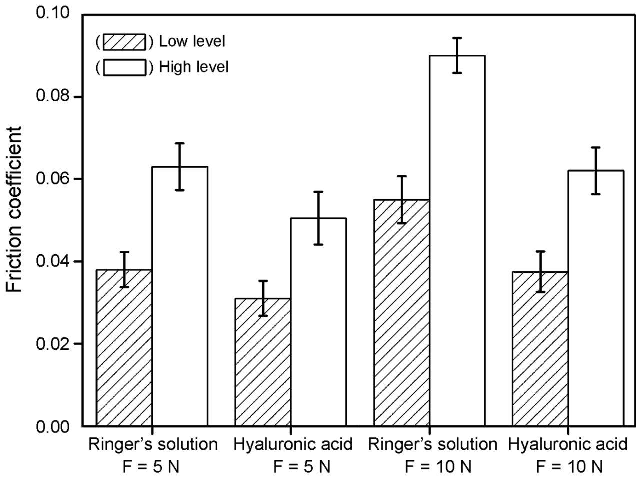


**Figure 5.** Effect of sliding speed on the friction coefficient; F is the load of each test.

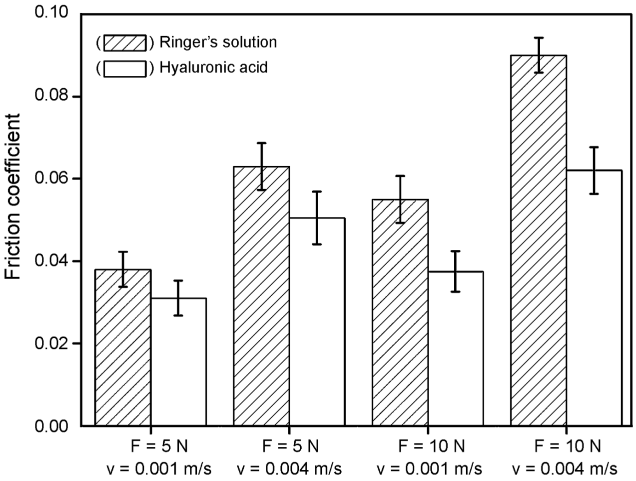


**Figure 6.** Effect of lubrication on the friction coefficient; F and v are the load and sliding speed of each test, respectively.

## Longer Term Friction Study

It can be observed from Figure 7 that the friction coefficient of PVA hydrogel against ceramic femoral component changed in 30 min. Friction measurements were taken at regular intervals from 25 s up to 1800 s. The frictional response produced gradual rising of the friction coefficient. This shows that loading time had significant effects on the frictional

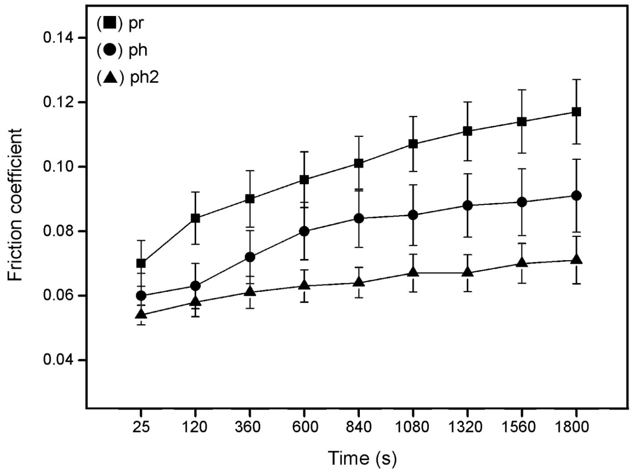


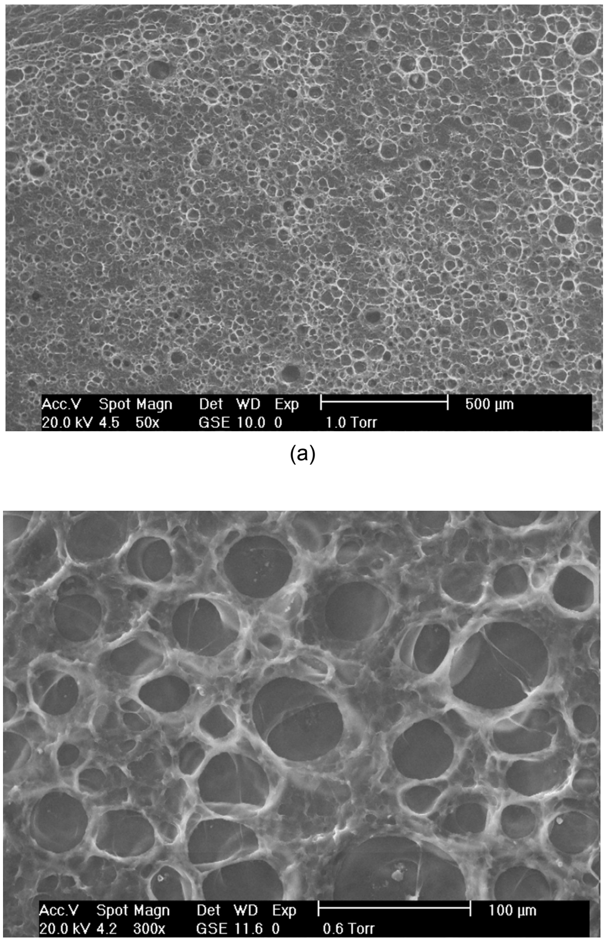
**Figure 7.** The change of friction coefficient in longer term friction tests. The abbreviations are as follows: pr, PVA hydrogel with RS as the lubricant under constant load; ph, PVA hydrogel with HA as the lubricant under constant load; ph2, PVA hydrogel with HA as the lubricant under cyclic load.

response. When the friction pair was lubricated by RS, the initial friction coefficient was close to that lubrication by HA. The incremental speed of the friction coefficient with HA lubrication is lower than that with RS lubrication. After 1320 s sliding tests, the friction coefficient under HA lubrication was 80 percent of the value recorded with RS. Analysis of variance of the friction coefficient also shows the effects of different loads. The friction tests under cyclical load resulted in a decrease in the friction coefficient compared with the constant load.

## Surface Analysis

The surface morphologies of unworn and worn PVA hydrogel under constant load in longer term tests examined by ESEM are shown in Figures 8 and 9, respectively. The ESEM examination of unworn PVA hydrogel reveals the porous structure in the specimen. There are no significant morphological features appeared on the surface. Figure 9 shows the wear surface of PVA hydrogel in 30 min constant load tests. Some scars and grooves are visible on the surface. The surface roughness alternations of PVA hydrogel plates are shown by the bar graph in Figure 10. When PVA hydrogel was lubricated by Ringer's solution, its surface roughness increased significantly compared to hydrogel lubricated by HA.

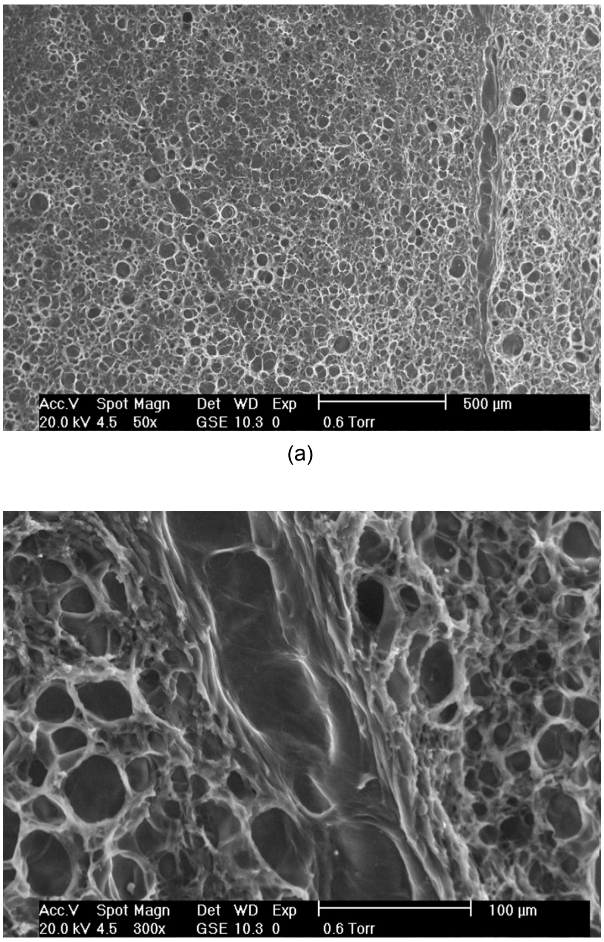
Since 1980, the "biphasic theory" was first put forward to explain the frictional property of articular cartilage [20]. The biphasic theory assumes the material to be made up of fluid phase and solid phase. The two phases are immiscible. Solid phase was a solid deformable porous matrix including collagen fibres, proteoglycans and other components. Fluid



(a)

(b)

**Figure 8.** ESEM images of unworn PVA hydrogel (a) and high magnification of the specimen (b).



(a)

(b)

**Figure 9.** ESEM images of worn PVA hydrogel (a) and high magnification of the specimen (b).

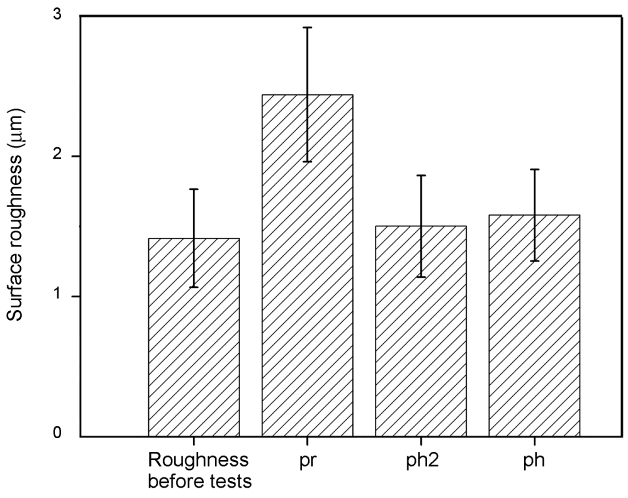


**Figure 10.** Surface roughness (μm) of PVA hydrogel before and after longer term tests. The abbreviations are as follows: pr, PVA hydrogel with RS as the lubricant under constant load; ph, PVA hydrogel with HA as the lubricant under constant load; ph2, PVA hydrogel with HA as the lubricant under cyclic load.

phase represents the interstitial fluid in its porous structure. Articular cartilage has very low permeability ranging from $10^{-15}$ to $10^{-16}$ $m^4/Ns$, thus the movement of the fluid through the solid phase induced high drag forces. The drag forces in articular cartilage help the fluid phase to withstand very high stresses. As a result, the solid phase withstands low stress and low level of solid to solid contact in the friction pair occurs. Then the friction coefficient of cartilage is kept low. Krishnan et al. [21] have found that when the rate of fluid loss from the contact zone increases, the intrinsic fluid pressure and load support by fluid phase are both reduced. When the solid to solid contact in the friction pair increases, the friction coefficient rises at the same time.

Then the biphasic theory was also used for other tissues and artificial materials, such as brain, meniscus and hydrogels [22,23]. Much like articular cartilage, PVA hydrogel has a three-dimensional structure with a large amount of water. Due to the matrix deformation and fluid transport through the porous structure, the biphasic theory is applicable to PVA hydrogel.

In the three-factor two-level designed experiment, the effects of different experimental factors on the friction coefficient between PVA hydrogel and ceramic femoral component are examined. It can be observed from Figure 4 that as load increases from 5 N to 10 N, the friction coefficient of each test increases as well. In Hertzian contact theory, the radius of contact circle is given as:

$$r = \sqrt[3]{\frac{3PR\left(\frac{1-\nu_1^2}{E_1}+\frac{1-\nu_2^2}{E_2}\right)}{4}} \tag{1}$$

Defined in eqn (1) as:

$$\frac{1}{R}=\frac{1}{R_1}+\frac{1}{R_2} \tag{2}$$

where $R_1$ and $R_2$ represent radii of curvature of ceramic femoral component and PVA hydrogel. Because the value approaches infinity, R is approximately equal to $R_1$. P is the load in the test. $E_1$, $E_2$, $\nu_1$ and $\nu_2$ are the elastic moduli and Poisson's ratios of ceramic femoral component and PVA hydrogel, respectively. PVA hydrogel has three-dimensional structure with a large amount of water and was more easily deformed than ceramic femoral component. The deformation depth of PVA hydrogel can be given as:

$$H = R_1 - \sqrt{R_1^2 - r^2} \tag{3}$$

when load P increased, the radius of contact circle r increased. Then, the increase of r induced the increase of deformation depth H of PVA hydrogel. When the load was applied, deformation in the surface of PVA hydrogel occurred. Because of the increase of deformation depth H of PVA hydrogel, a larger amount of fluid in PVA hydrogel was extruded from the porous structure under high level applied load. Then the solid contact area between ceramic femoral component and PVA hydrogel increased, thus the solid part of PVA hydrogel supported greater proportion of the applied load and the friction coefficient significantly increased.

In the previous research work by Pan et al. [12], PVA hydrogel sample rotated under the static titanium alloy ball with high speed ranging from 0.06 m/s to 0.22 m/s. Pan et al. observed that as the sliding speed increased, the friction coefficient decreased. In this study, low speed of 1 mm/s and 4 mm/s was used to ensure the contacts maintained within a mixed or boundary lubrication regime. It is inferred that when the speed is low, the friction coefficient increases with the increases in speed. In the experimental study by Pan et al., the samples were tested in air and water. It was shown that the friction coefficient of PVA hydrogel has no obvious difference under dry and water lubricating conditions. Most solid materials conform to Amonton's law which means that the frictional coefficient is only proportional to the applied load. In comparison to frictional behaviour of solid material, such as glass or metal, the friction coefficient of PVA hydrogel highly depends on the sliding speed. It can be concluded that Amonton's law is not suitable for PVA hydrogel. When the sliding speed was low, the recovery of fluid phase in the reciprocating motion was long and the recovery was more efficient. In the recovery time, the fluid phase was absorbed to the porous structure of PVA hydrogel and the surface deformation of PVA hydrogel decreased. As a result, the friction coefficient of the friction pair kept low. At high level of sliding speed, the complete recovery of PVA hydrogel could not complete. Surface deformation of PVA hydrogel in high speed was larger than that in low speed. The solid phase withstood higher stress and the friction coefficient increased.

Covert et al. [11] studied the frictional behaviour of PVA hydrogel against stainless steel. It was shown that dynamic friction was dependent on sliding speed, but the load effects were not significant. In our study, the dynamic friction was dependent on both load and sliding speed. Gong et al. [24] proposed that the frictional force could be expressed as functions of applied normal load, sliding speed, polymer volume fraction and elastic modulus of hydrogel. They found that when sliding speed is not very large, the friction of hydrogel exhibits a large dependency on the speed. The theoretical results show a linear relation between frictional forces and sliding speed. The theoretical results also coincided well with their previous experimental results [25]. In this study, the frictional force increased while the applied load and sliding speed increased. The results in this study agreed well with theoretical results of their hydrogel model.

In recent years, HA has been introduced for injection into the joints to treat osteoarthritis (OA) of the knee. Intra-articular hyaluronan injections for the treatment of OA patients could have many benefits, including pain relief and function improvement of their joints. HA has high viscosity and water-retention properties [26], which have several effects on lubrication. In this study, the friction coefficient of PVA hydrogel against ceramic femoral component decreased significantly with lubrication condition of HA. The friction coefficient at HA lubrication was almost half of that at Ringer's solution lubrication. When the load and speed are at high level, HA lubrication shows more effects on the friction coefficient.

In longer term friction tests, the increase rate of the friction coefficient under cyclic load is marked lower than that in continuous sliding tests (Figure 7). At 1800 s, the friction coefficient of the cyclic load was 0.071 which was almost two thirds of the value in constant load tests. In the constant load tests, large amount of the fluid phase in PVA hydrogel was extruded from the porous structure and most loads were supported by the solid phase in PVA hydrogel. When the interstitial fluid phase was continuously lost, the porous structure of PVA hydrogel withstood high level of stress and the surface wore off. The surface failure of worn PVA hydrogel was observed by ESEM. The wear of PVA hydrogel could also be identified by the change of surface roughness. In contrast, the cyclic load encouraged the rehydration of the fluid phase. Then the fluid phase could maintain its load support by rehydration, thus the friction coefficient of the cyclic load tests increased very slowly. It is indicated that the biphasic property of PVA hydrogel has great effect on the friction property of the friction pair.

## CONCLUSION

In three-factor and two-level designed experiment, applied load, lubrication and sliding speed had significant effects on the friction coefficient of PVA hydrogel against ceramic femoral component. The results showed that when sliding speed is low, the friction coefficient increased with the increase of sliding speed. HA was an effective lubricant in all tests.

In longer term friction tests, lubrication condition and friction time employed had significant effects on the friction behaviour. The friction coefficient of the cyclic load tests was much lower than that of the constant load tests. The wear of PVA hydrogel under constant load was identified by ESEM. It can be concluded that the biphasic lubrication property is the key feature for PVA hydrogel. Further studies are still necessary to optimize the biphasic performance of PVA hydrogel.

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