

Supporting information

In-situ fabrication of carbon dots-based lubricants using a facile ultrasonic approach

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Experimental Section

Materials

Citric acid (CA, AR grade), urea (AR grade) and PEG with an average molecular weight of 380–420 g mol⁻¹ (AR grade) were purchased from Tianjin Kermel Chemical Reagent Co., Ltd. (China). Dichloromethane (DCM, AR grade) was obtained from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All chemicals were used as received without further purification.

Instrumentation

Ultrasonic treatment was achieved using a 70 W ultrasonic cleaner (Kun Shan Ultrasonic Instruments Co., Ltd. China). High resolution transmission electron microscopy (HRTEM) images were taken with a Tecnai F20 transmission electron microscope (FEI, USA). Raman spectra were characterized using a DXR microscope Raman spectrometer (Thermo Fisher, USA). Fourier transform infrared (FT-IR) spectra were confirmed by a Nicolet-6700 FTIR spectrophotometer (Thermo, USA). UV-Vis absorption spectra were acquired using a Lambda 750 UV-Vis-NIR spectrophotometer (Perkin Elmer, USA). Photoluminescence (PL) spectra were measured by an F-7000 fluorescence spectrophotometer (Hitachi High Technologies, Japan). X-ray photoelectron spectroscopy (XPS) was carried out by a X-ray photoelectron spectrometer (Thermo, USA) with an Al K α 280 eV radiation source and the C1s peak at 284.8 eV as a binding energy calibration.

Tribological property measurements

The friction and wear tests were conducted on a MMW-1A microcomputer control friction and wear tester (Jinan Yihua Tribology Testing Technology Co., Ltd. China) through a four-ball model under the following conditions: rotating rate, 1200 rpm; duration, 60 min; load, 600 N; and temperature, 75 °C. In detail, an AISI52100 steel ball with 12.7 mm in diameter and hardness of 59–61 HRC was rotated over three identical balls, as shown in Fig. S1A. Three-dimensional (3D) images and surface roughness of lower balls were confirmed by an OLS4000 confocal laser scanning microscopy (Olympus, Japan), and friction coefficients were recorded

simultaneously by a computer linked to the tester. Scanning electron microscopy (SEM) images were obtained using an FEI-quanta 450 scanning electron microscope (FEI, USA) equipped with Energy Dispersive X-ray Spectrometer (EDS) analyses.

Synthesis of CDs-based lubricants

In a typical synthesis, 0.104 g solid mixture of CA and urea with mass ratio of 1:1 was added into 12.896 g PEG at room temperature under ultrasonic treatment for 1 h. Then the 13 g CDs-based lubricant with concentration of 0.8 wt% was obtained. Then the CDs-based lubricant with concentration of 0.8 wt% was obtained and named as 0.8 wt% CDs-1:1, where 0.8 wt% and 1:1 represent the mass ratio of solid mixture/PEG and CA/urea, respectively. For mass production of the CDs-based lubricant, we proportionally increased the amount of CA, urea and PEG. Under the existing experimental equipment, the CDs-based lubricant with content of 0.8 wt% could be prepared at the rate of 1 kg/h. It should be easy to fulfill larger scale fabrication if more powerful ultrasonic equipment was employed. Furthermore, through altering the molar ratio of CA, urea and PEG, the CDs-based lubricants with different tribological properties could be prepared. In addition, more measures, such as hydrothermal process (75 °C) and stirring process, were also explored to produce CDs-based lubricants instead of ultrasonic treatment.

Separation of solid-state CDs from CDs-based lubricants

To obtain the solid-state CDs from CDs-based lubricants for UV-Vis spectra, PL spectra, Raman spectra, FT-IR spectra and XPS, solvent extraction was adopted. In detail, the CDs-based lubricant was firstly dispersed in DCM. Then deionized water was added. After stirring, the DCM phase containing lots of CDs and limited PEG was separated using a separatory funnel and the water phase including lots of PEG was removed. Subsequently, repeat the extraction process until the PEG was fully removed. Afterwards, the DCM phase containing lots of CDs was transferred into an oven to eliminate DCM. Finally, faint yellow solid-state CDs were obtained. The photograph of solid-state CDs is shown in Fig. S1B.

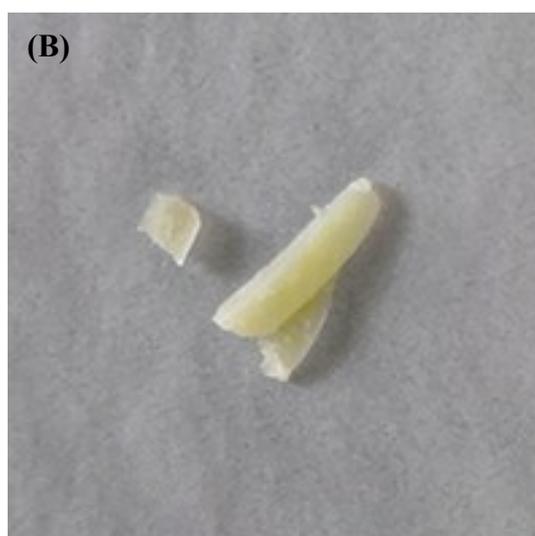
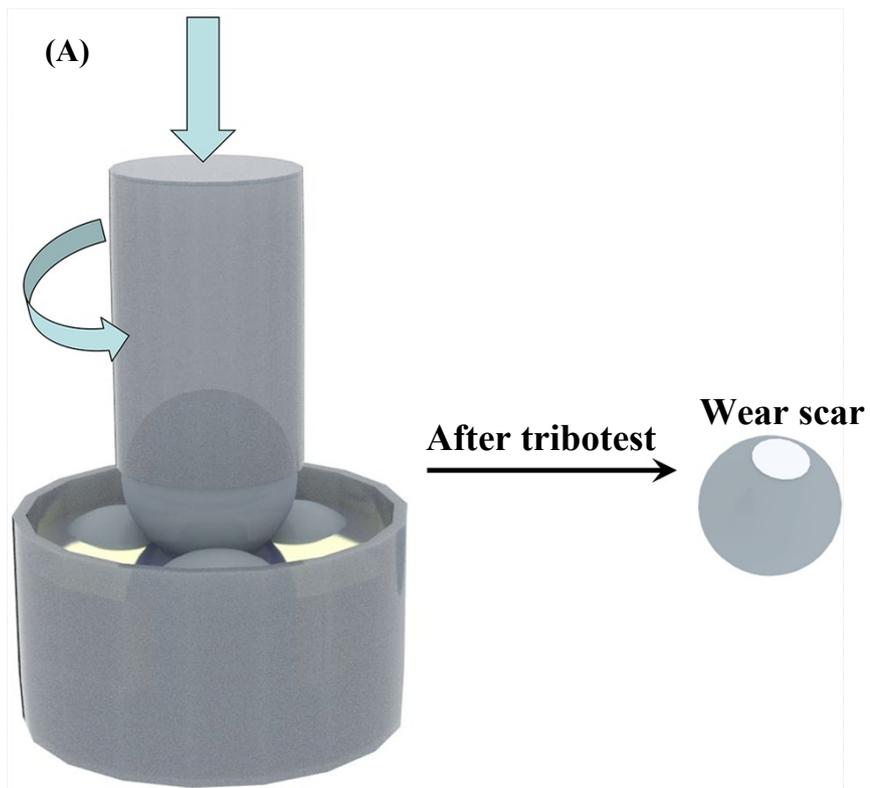


Fig. S1 (A) Schematic of the four-ball model; (B) The photograph of solid-state CDs taken under sunlight light.

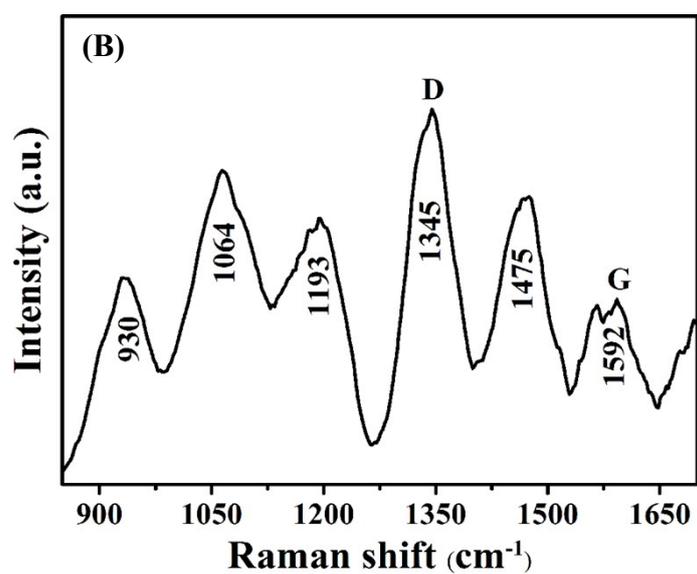
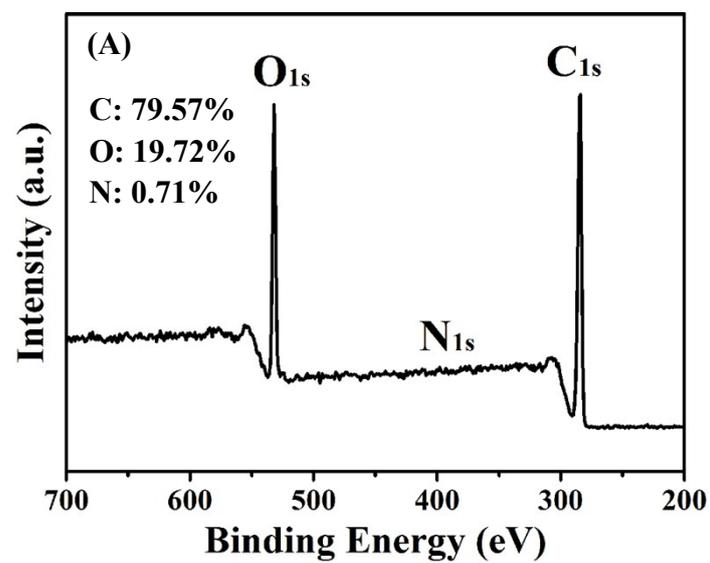


Fig. S2 (A) Full range XPS spectra and (B) Raman spectroscopy of CDs.

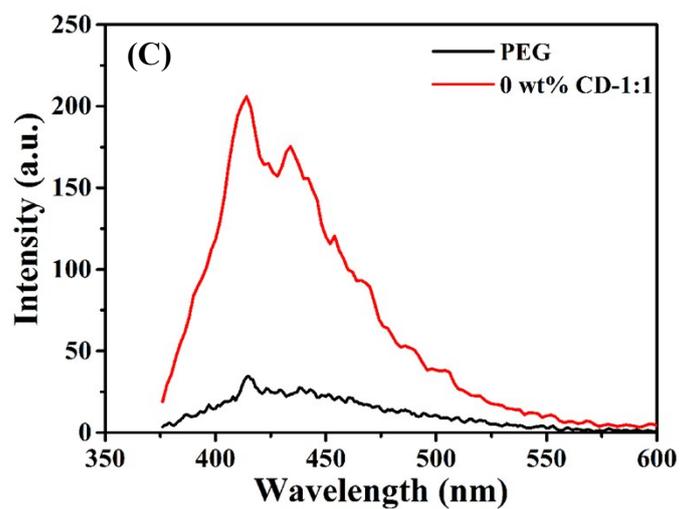
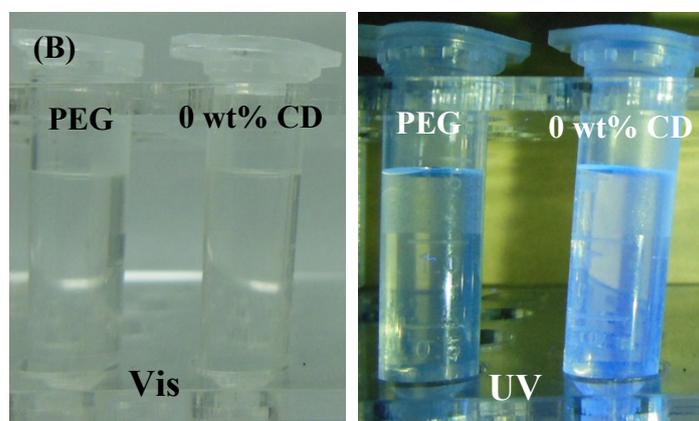
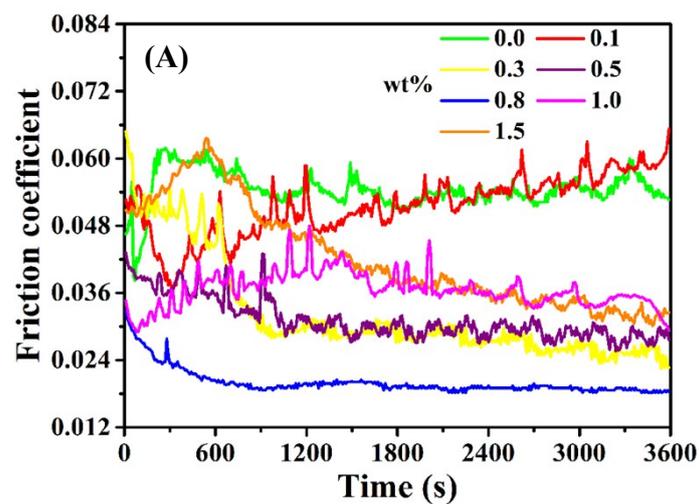


Fig. S3 (A) The friction coefficient curves of c wt% CDs-1:1 (load: 600 N; speed: 1200 rpm; duration: 60 min; temperature 75 °C); (B) Photographs of PEG and 0 wt% CD-1:1 under visible and UV light and (C) their PL spectra under 365 nm excitation.

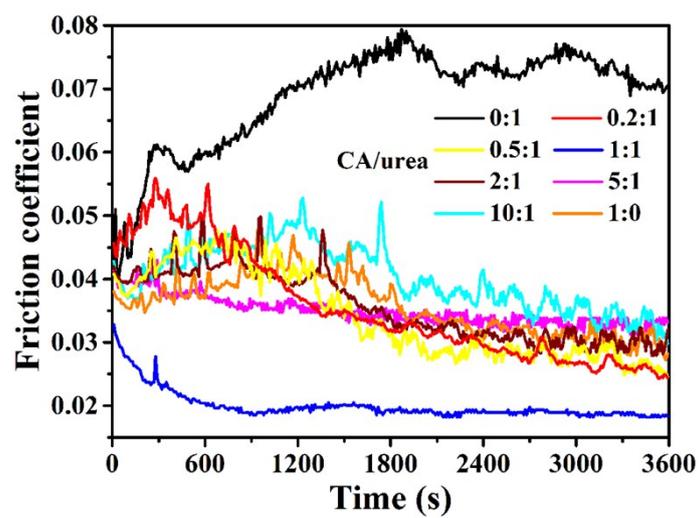


Fig. S4 The friction coefficient curves of 0.8 wt% CDs-m (load: 600 N; speed: 1200 rpm; duration: 60 min; temperature 75 °C).

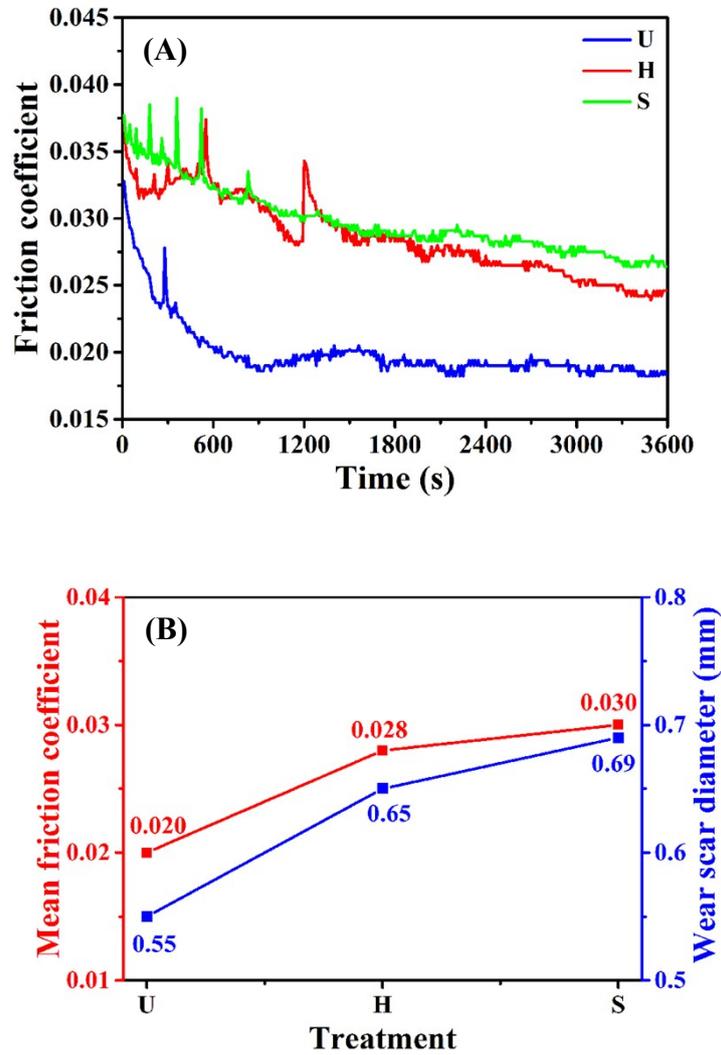


Fig. S5 (A) The friction coefficient curves of CDs-based lubricants at 0.8 wt% concentration treated by ultrasonic (U), hydrothermal (H) and stirring (S) processes (load: 600 N; speed: 1200 rpm; duration: 60 min; temperature 75 °C); (B) The mean friction coefficient and wear scar diameter corresponding to U, H, S processes.

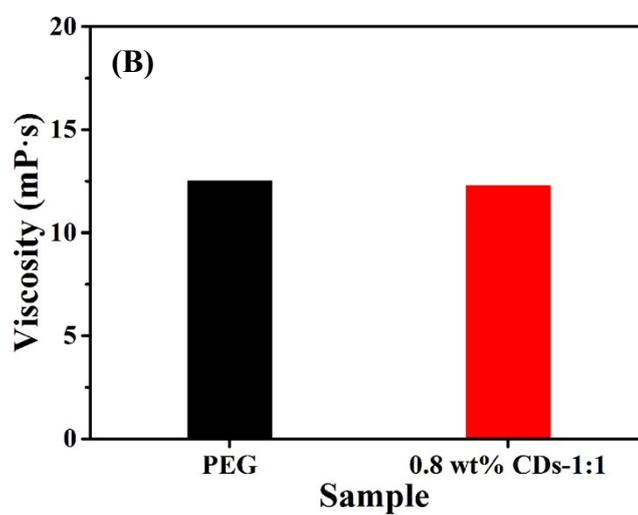
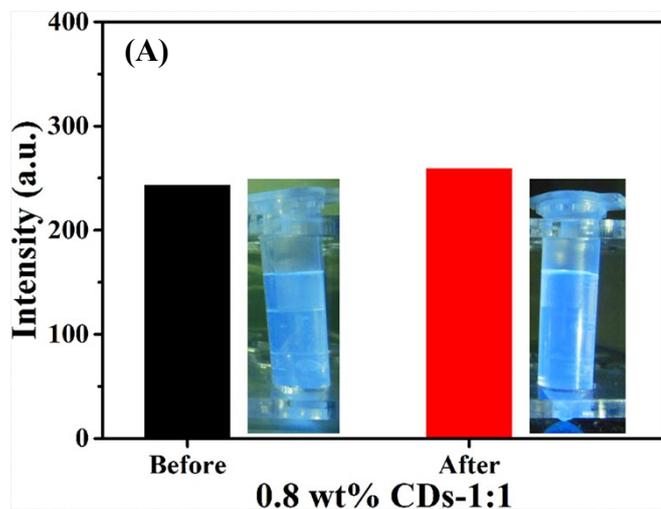


Fig. S6 (A) PL intensity and photographs of 0.8 wt% CDs-1:1 under 365 nm excitation of before or after tribotest; (B) The viscosity at 75 °C of PEG and 0.8 wt% CDs-1:1.

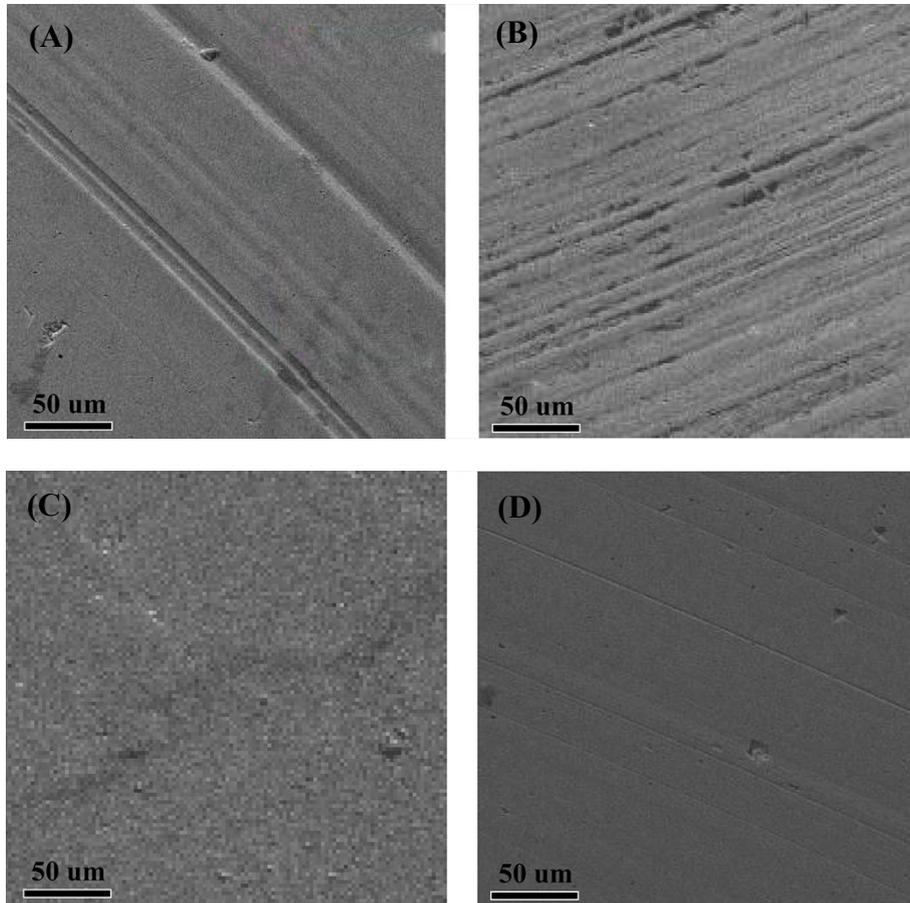


Fig. S7 SEM images for center area of the worn surfaces of lower steel balls: (A) PEG; (B) 0 wt% CD-1:1; (C) 0.8 wt% CD-1:1; (D) 1.5 wt% CD-1:1.

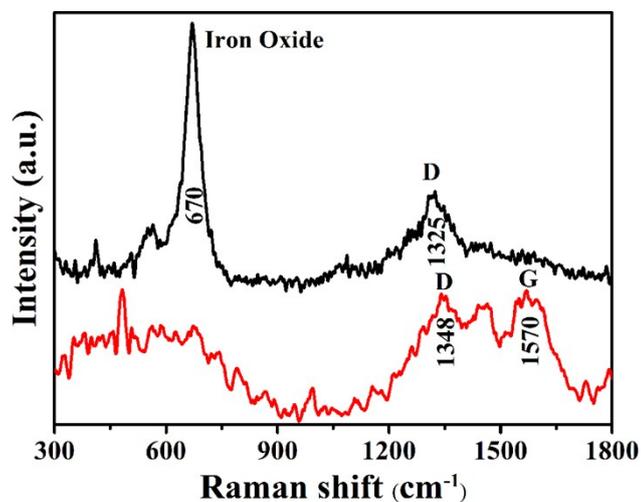


Fig. S8 Raman spectra of worn surfaces lubricated by PEG and 0.8 wt% CD-1:1.

Table S1. Elemental compositions on the worn surfaces of lower steel balls lubricated by PEG and 0.8 wt% CDs-1:1 lubricant under the load of 600 N.

Lubricant	C (%)	O (%)	Fe (%)	Mn (%)	N (%)
PEG	38.18	18.25	43.36	0.21	0
0 wt% CDs-1:1	37.89	17.17	44.71	0.23	0
0.8 wt% CDs-1:1	51.75	6.50	41.53	0.22	0
1.5 wt% CDs-1:1	35.99	3.29	60.42	0.29	0