Fracture Behavior of Wasted Activated Carbon Powder Composites

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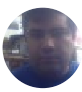

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At the Waterworks Bureau (Tokyo Metropolitan Government), activated carbon has been used for filtering water. After being used for the filtering process, it is normally disposed or burned for thermal recycling. However, CO_2 emissions occur during the thermal recycling. This work focuses on the identification of mechanical behavior of recycled wasted activated carbon (WAC) in order to elaborate smart materials having mechanical–electrical functions. Acoustic emission technique (AE) was used intensively as characterization support in which sensors were attached to detect microdamage during bending tests. At first, the resonant frequencies of the specimens were measured using the through-transmission test. The resonant frequencies of the specimens containing low weight fractions of WAC powder were less in comparison to the frequencies of the specimens with higher volume fraction. The frequency analysis was carried out with the projected wavelet transform on the signals detected during bending tests. Obtained data showed that, typically, the first major peaks showed the resonant frequency of the sensors, while the second major peaks exhibited signals indicative of resin cracking. The surfaces of the fractured specimens were analyzed by optical microscopy in order to visualize the crack formation and propagation on the activated carbon composite under flexural stresses. Consequently, fractographic and AE analyses provide better understanding of the failure mechanisms involved.

Keywords: polypropylene; wasted activated carbon powder; mechanical property; damage behavior; acoustic emission

1. Introduction

Tokyo Metropolitan Government Bureau of Waterworks has planned to make 'advanced clean water processing facilities' in which activated carbon particles have been used for 'biological activated carbon treatment.' Activated carbon has been processed to make an extremely porous material and thus has a very large surface area available for adsorption or chemical reactions. Due to its high degree of microporosity, just one gram of activated carbon has a surface area in excess of 500 m^2.[1,2] After using the activated carbon particles for the biological activated carbon treatment, the material is burned and/or buried to avoid its contribution to the greenhouse effect as

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CO_2. It is, therefore, very important to acknowledge the need of understanding how the particles of the wasted activated carbon (WAC) should be recycled and reused while minimizing any threat of environmental pollution. On the other hand, activated carbon powder has been lately considered as an important material used as filler in composites due to its excellent adsorption for controlling toxic air pollutants and for conferring some electrical properties to some materials such as polymers.[1]

Recently, the electrical properties of carbon nanofiber composites have been investigated showing excellent conducting characteristics. Furthermore, thermal, mechanical, and electrical properties of copper powder-filled polymer were also explored and they showed good electrical property and low crystallinity, low glass transition temperature and low-mechanical properties. The activated carbon–polymer-based composites have demonstrated great acceptance in the electrical and electrochemical fields.[3–6] The electrical resistance is determined by the connectivity of the conducting particles in the nonconducting matrix, and therefore, the relative amount of each constituent has to be assessed to achieve optimal composition. WAC particles are also expected to exhibit electrical properties, so the electrical properties and electrical conductivity of wasted carbon particle composites were studied in this research.[7]

Using the electrical conductivity of WAC powder composites as a parameter, new smart materials from recycling of wasted carbon can be developed.[7] In order to use these materials for manufacturing smart composites using its electrical properties, it is necessary to understand the mechanical and damage behavior of the materials. To investigate the damage behavior, the acoustic emission (AE) technique was adopted to follow the sequence of failure until total fracture. The AE technique is used for detecting microcracking, debonding, and fiber breaking in composite materials.[8–10] To detect the microcracks, researchers used not only PZT sensors, but also FBG sensors.[11,12]

The purpose of this study is to understand the mechanical properties by identifying the microdamage behavior and failure sequence of the WAC powder composites. The mechanical properties and the damage accumulation behavior of the composites were investigated by static bending tests with the AE technique. Consequently, the fracture modes of the composites, which were made with WAC powder and polypropylene, were identified by frequency analysis with a projected wavelet analysis.

2. Materials, processing, and experimental condition

2.1. Materials

In this study, the WAC particles, which had been used in the Bureau of Waterworks Tokyo Metropolitan Government, were used as filler for manufacturing composite specimens. They have fewer pores than normal activated carbon particles because some of their pores were filled with inorganic materials when used initially for water filtration.

Polypropylene MA3 (PP; Japan Polypropylene Co., Ltd) was employed as the polymeric matrix. The melting point and glass transition temperature for PP are 150 and 127 °C, respectively, as determined by differential scanning calorimetry analysis.

2.2. Processing

In order to use the WAC particles as filler in the composite specimens, they were crushed with a ball mill for 8 h. Crushed particles were then sieved in order to obtain a uniform size powder with grain size of approximately 8 μm. PP pellets were dried for

24 h in an oven at 70 °C before mixing to avoid humidity in the blends. Blending was carried out in a mixing chamber 1882 (Imoto Machinery Co., Ltd) with 64 cc capacity. Figure 1 illustrates the mixing and lamination processes.

The composite material obtained from the mixing machine is completely amorphous and homogeneous. A good dispersion of the WAC filler in the polymer matrix (PP) was observed.

The composite blend was pressed in a mold to create laminae containing 10–60 wt% WAC powder for mechanical and electric characterization. Coupons were obtained from these laminae for mechanical testing. After molding the materials, they were quenched with cold water.

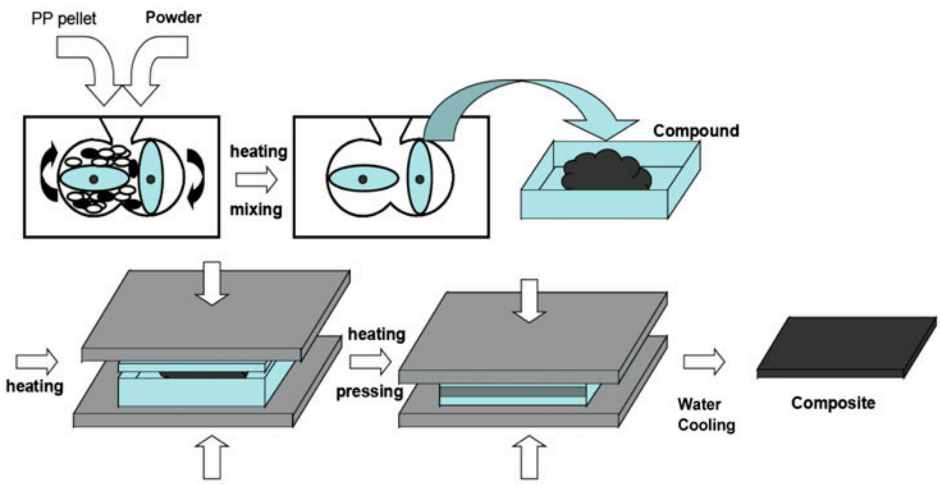

Figure 1. Schematic drawing of blending of the composite and lamination.

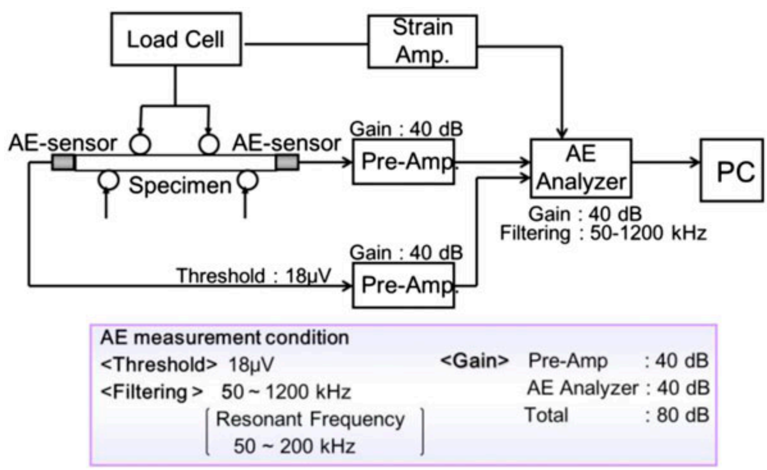

Figure 2. Bending testing systems with AE technique.

2.3. Experimental conditions

Bending tests were carried out on 3 × 4 × 40 mm specimens with a universal testing machine (Auto Graph, Shimadzu Co., Ltd) in air atmosphere at a cross-head speed of 0.5 mm/min, with symmetrical loading points at 30 and 10 mm from one end.

The AE technique was used to monitor the damage development in the samples during the bending tests. Figure 2 shows the AE measurement system for the four-point bending test, with the key parameters and settings included. Broadband-type AE sensors (AE-900M, NF Corp.) and AE analyzer AMSY-5 (Vallen Systeme GmbH) were employed in online damage monitoring. To detect the cracks and to reduce noises from outside, the specimen using the sound wave velocity and the detected time differential, two sensors were attached to the specimen, one at each end.

3. Mechanical properties

3.1. Bending properties of materials

Figure 3 shows the bending strength and moduli of the WAC/PP composites. Bulk PP (0 wt%) specimens failed with no complete fracture exhibiting about 34 MPa. For composites containing 10–50 wt% WAC, the bending moduli is only slightly higher than bulk PP (0 wt%), while the 60 wt% composite possesses the highest value. Therefore, for the 60 wt% specimens, it seems the WAC powder dominates the bending modulus, but the matrix resin dominates this property for lower powder contents.

Conversely, the bending strength of 20, 30, and 60 wt% specimens is higher than the others, with 20 and 30 wt% close to 65 and 68 MPa, respectively. This suggests that the powder effectively reinforced the matrix for 20 and 30 wt% specimens, whereas the higher powder content in 60 wt% specimens made the matrix resin act more as adhesive between particles rather than a bulk material.

3.2. Resonant frequency of materials

It is necessary to take into account the characteristic frequency response of the sensors and the specimens tested when considering the result of the AE wavelet analysis. In order to evaluate this response, the through-transmission test system shown in Figure 4

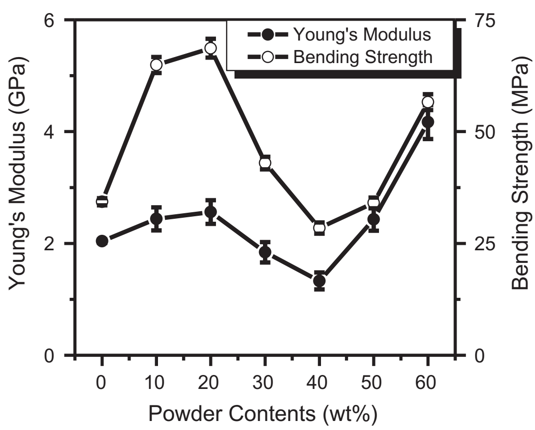

Figure 3. Bending strength and moduli of WAC powder composites.

was used. The frequency response of the sensors and specimens is estimated in Figure 4(a), and the response of the sensors alone is assessed in Figure 4(b). Therefore, if the value measured in Figure 4(b) is divided by the value measured in Figure 4(a), the frequency response of the specimens can be calculated. Function generator SG4115 (IWATSU Co., Ltd) and AE analyzer AMSY-5 (Vallen Systeme GmbH) were used in this evaluation. Generated waves were changed from 50 to 1200 kHz, and the tone burst waves were used for the input signals.

Figure 5 shows the results of the first-peak resonant frequency measurement by the through-transmission tests. Increasing the powder content causes augmentation in the resonant frequency. The resonant frequencies of the specimens with over 40 wt% WAC powder are higher than those with under 30 wt%. Over 40 wt%, the materials became denser with the WAC powder, therefore, the high-frequency components of the waves were transmitted.

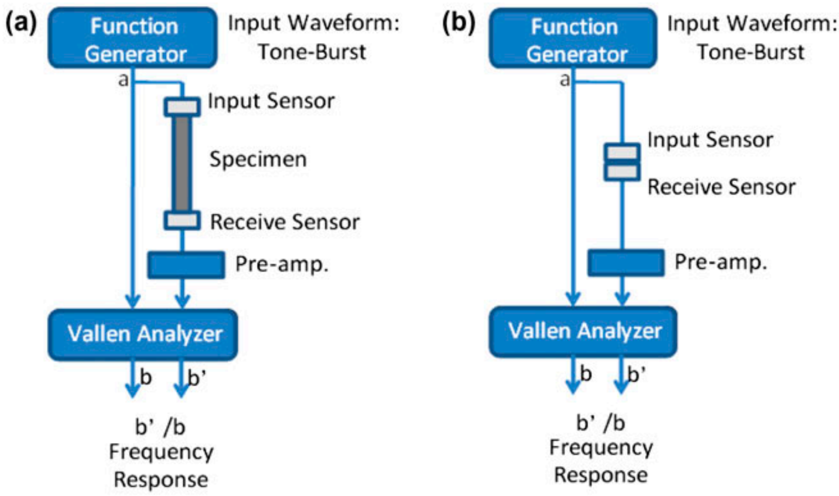

Figure 4. Schematic drawing of through-transmission testing systems (a) through the specimen and (b) without specimen (sensor's response).

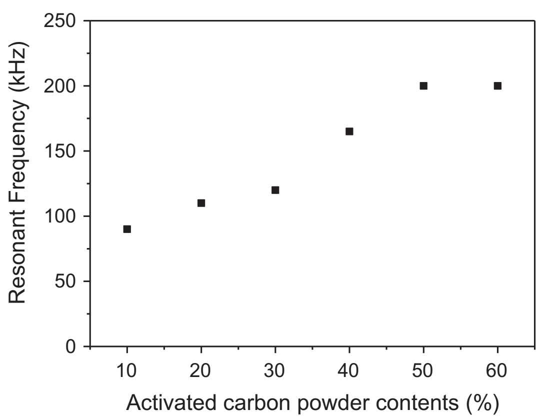

Figure 5. Frequency responses of each WAC powder composites.

3.3. Mechanical and damage accumulation behavior of composites

Figure 6 shows the result of bending tests of the 60 wt% sample with the acoustic signals detected (amplitude and energy). AE counts were first observed to rise (around 75 s, on the dotted line), when a bending crack (shown in Figure 7, on a chain line) initiated, and then the load-time curves came off from the linear relationship.

To understand the frequency characteristics of waves detected during bending tests, the projected wavelet transform analysis is adopted to classify the AE signals in terms of the transient frequency component.[13,14] Figure 8 illustrates the analysis procedure. Analyzing the frequency at the peak wavelet coefficient, which indicates strength at given times and frequencies in a signal, the first and second major frequencies of each AE signal are extracted. In the case of the first major frequency, it will show the

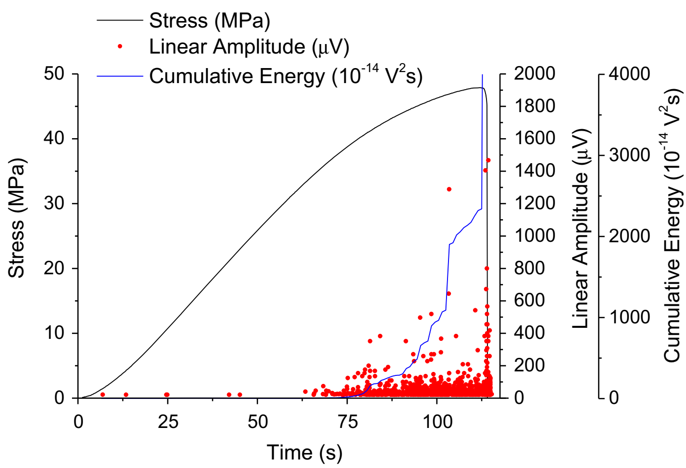

Figure 6. Bending behavior with acoustic information of 60 wt%.

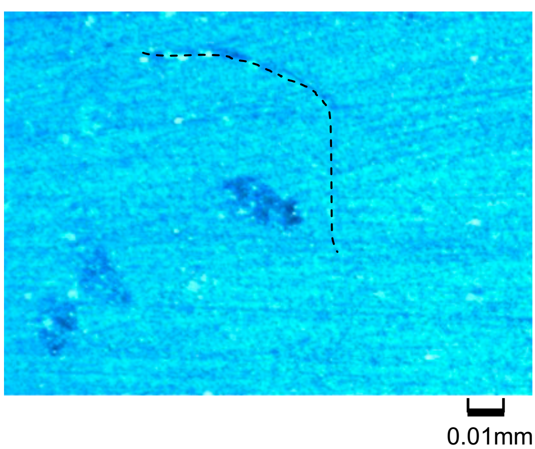

Figure 7. Observed crack when AE energy first increased (around 75 s in Figure 5) (60 wt%).

resonant frequency of the AE sensors or the distinguished frequency of the crack initiation and propagation. In the case of the second major frequency, it will show the distinguished frequency of the crack initiation and propagation.

Figure 9 shows the history of the first (red circle) and second (blue circle) major frequencies of the specimens of each carbon powder weight fraction, which were obtained during the bending test, and the relative values of the wavelet transforms

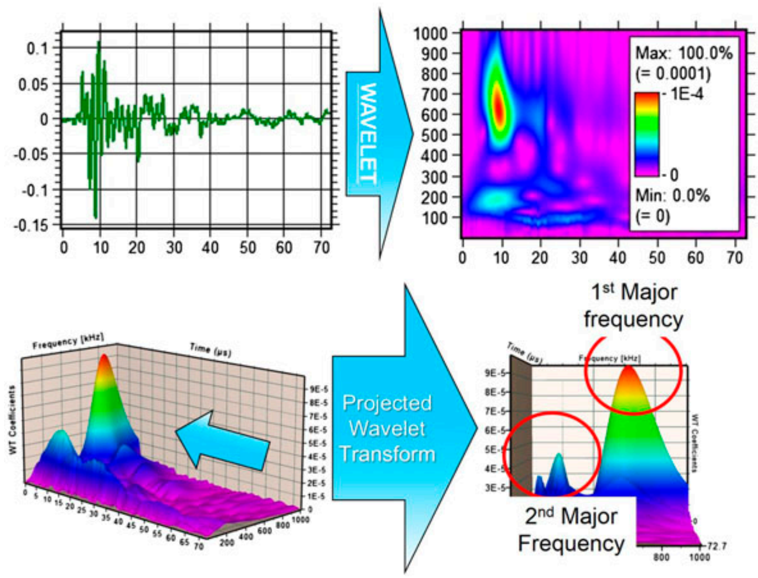

Figure 8. Schematic drawing of wave analyzing with projected wavelet transform method.

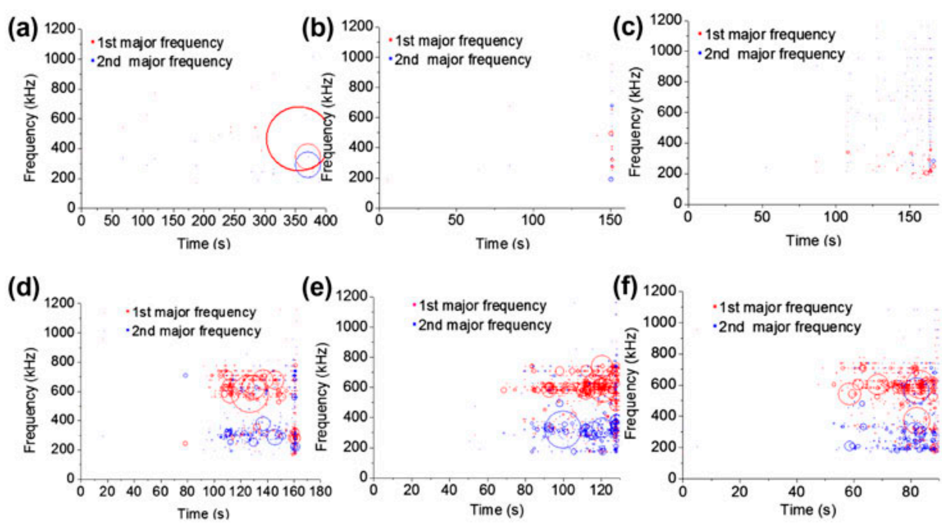

Figure 9. First and second major frequencies of AE signals detected during bending tests of (a) 10%, (b) 20%, (c) 30%, (d) 40%, (e) 50%, and (f) 60%.

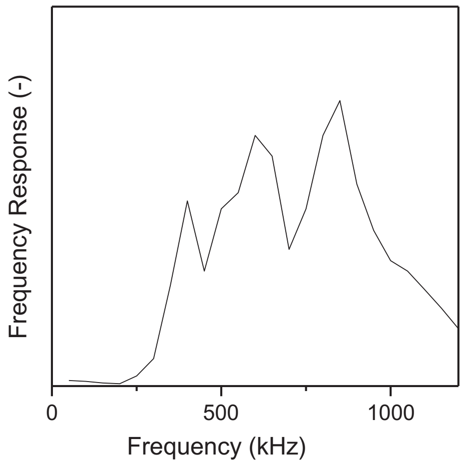

Figure 10. Frequency response of used AE sensor.

coefficients of the first and second major frequency components are indicated by the circle diameter. Here, AE signals were not detected for the bulk PP specimens. As shown in Figure 9(a)–(c), fewer signals were detected than in Figure 9(d)–(f), since the density increases enough with over 40 wt% WAC powder for the waves to transmit clearly.

Two major frequencies were observed. The first major frequency components are distributed in a limited frequency band of 500–700 kHz, and the second major frequency components are distributed in 250–400 kHz.

In order to consider the frequency band of 500–700 kHz, the sensor's frequency response was measured by the through-transmission tests, because the first major frequency will be affected by the sensor's resonant frequency. Figure 10 shows the frequency response of the AE sensors used in this study. As shown in this figure, the sensor's resonant frequencies are around 850 and 600 kHz. The lower frequency as 600 kHz corresponds to the first major resonant frequency of the detected AE signals during tests; therefore, the first major frequency would be affected by the sensor's frequency response.

In the case of the second major frequency, it is the same frequency (250–300 kHz) as identified before by Arumugam et al. [15], which tends to show matrix cracking occurring in the composite. In the present materials, it is considered that the WAC particles became the initiators of the microcracks, as spherical inclusions in the polymer matrix.

The surfaces of the fractured specimens were analyzed with optical microscopy under polarized light in order to visualize the crack formation and propagation on the activated carbon composite under flexural stresses. Figure 11(a) displays the fracture surface and Figure 11(b) shows the manner in which samples were fractured by both tensile and compression stresses, given the typical vertical crack shape. Figure 11(a) shows the aerial view on the fractured surface where it is possible to observe that around the compression area of the specimen, a smooth surface is visible, and in the tension zone the surface was rough due to the successive cracks that appeared when the materials was submitted to high levels of stress during the flexural test. Such fracture behavior was observed in all materials tested.

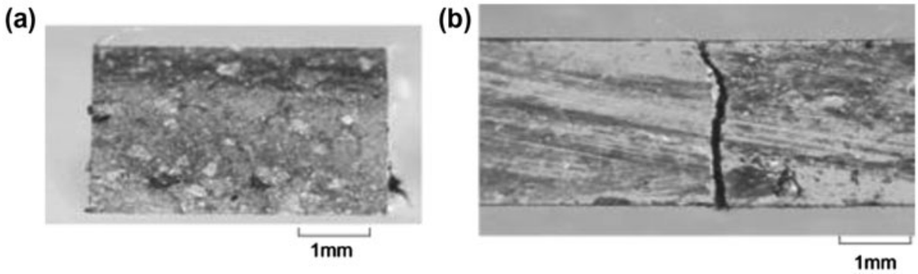

Figure 11. (a) Fracture surface and (b) side face for WAC composite of 50 wt%.

In Figure 11(b), compression zone was characterized by a sharp point fracture, whereas the tension zone exhibited clear straight separation. This fracture mode was observed in all samples tested. On the whole, all the samples invariably exhibited fracture modes as described before. Supported by the fractographic analysis and AE technique, the failure mechanisms suggest the following progression during the bending test:

(1) microdamage initiates in the tensile region of the transverse surface;
(2) microcracks appear at low strains, identified by low amplitude and energy;
(3) at higher strain (generally after 450 s), larger matrix cracks in the tensile region appear, increasing the amplitude and energy of the AE signals;
(4) the WAC/PP composite behaves similarly to thermoplastic, showing good plasticity that allows the development and propagation of matrix cracks through the sample;
(5) Finally, the material experiences a sudden fracture.

4. Conclusions

The fracture behavior of the activated carbon powder reinforced polypropylene was investigated through static bending tests. The following conclusions were made:

(1) The resonant frequency of the specimens increased with the WAC weight fraction.
(2) During bending tests, AE signals were detected on the specimens over 40 wt%, because of their higher density.
(3) The microcracking of matrix resin occurred during the bending tests as detected by the AE technique.
(4) Fractographic and AE analyses were effective techniques for understanding the fracture behavior of WAC powder composites during static bending tests.

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