Effect of powdered activated carbon technology on short-cut nitrogen removal for coal gasification wastewater

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HIGHLIGHTS

- The PACT and SBNR combined system enhanced the TN removal efficiency for CGW.
- The SBNR performance was positively related with PACT removal efficiency.
- The PAC removed some recalcitrant compounds, which was indispensible for SBNR.

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Abstract

A combined process consisting of a powdered activated carbon technology (PACT) and short-cut biological nitrogen removal reactor (SBNR) was developed to enhance the removal efficiency of the total nitrogen (TN) from the effluent of an upflow anaerobic sludge bed (UASB) reactor, which was used to treat coal gasification wastewater (CGW). The SBNR performance was improved with the increasing of COD and TP removal efficiency via PACT. The average removal efficiencies of COD and TP in PACT were respectively 85.80% and 90.30%. Meanwhile, the NH$_3$–N to NO$_2$–N conversion rate was achieved 86.89% in SBNR and the total nitrogen (TN) removal efficiency was 75.54%. In contrast, the AOB in SBNR was significantly inhibited without PACT or with poor performance of PACT in advance, which rendered the removal of TN. Furthermore, PAC was demonstrated to remove some refractory compounds, which therefore improved the biodegradability of the coal gasification wastewater.

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1. Introduction

Discharge of coal gasification wastewater (CGW) wastewater would cause significant deterioration of the environment due to the presence of nutrients especially hazardous substances and persistent compounds, for its high strength in refractory organics and ammonia (Gai et al., 2008; Wang et al., 2011a,b). CGW treatment is the most difficult pollution control task in the coal chemical industry. Regardless of economic value, ozonation, wet air oxidation (Freese and Rolinski, 1984), Fenton-coagulation (Liu et al., 2004) had been effectively to used to remove organic compounds, in terms of BOD$_5$, NH$_3$–N, volatile phenol and chroma. In contrast, activated sludge system was cost-effective over the physiochemical methods for less energy or chemicals required.

According to the reports, two-stage anaerobic reactor has been successfully applied as first step in the full-scale treatment plant (Wang et al., 2011a). With it, COD and total phenol could be remarkably removed, especially after employed optimal feeding mode and proper co-substrate (Wang et al., 2010, 2011b). But the ammonia nitrogen, as well as the remaining COD, phenol, other pollutants which exhibit poor removal efficiency in anaerobic process needed to be removed by the followed aerobic treatment in order to meet the requirement of concerned standard. However, the conventional activated sludge may not so effective to accomplish the biological nitrogen removal of CGW. Li et al. (2011) found that nitrification in MBBR would be easily inhibited especially during the start-up period or sudden increasing of COD or NH$_3$–N in the CGW influent. Similarly, Ramos et al. (2007) used submerged fixed-film reactor to accomplish biological nitrogen and phenol removal from saline industrial wastewater and found that this process was not able to achieve a nitrogen oxidation superior to 63%, in spite of a sufficient supply of oxygen and the diluting effect of high recirculation (600%) on the phenol concentration in the influent. One reason for the low efficiency of total nitrogen removal was attributed to the harmfulness and toxicity of CGW. Typical nitrification inhibitors, such as phenol, polynuclear aromatic hydrocarbons, and nitrogen hetero-
clic compounds resulted in insufficient nitrification. Another explanation could be inadequate carbon resource for denitrification. The COD/N value was only an apparent overall indicator; the stoichiometric parameter is in fact the denitrification potential of wastewater, i.e. the fraction of organic carbon available for denitrification (Hocaoglu et al., 2011). Therefore, due to the distinct characteristic of CGW, problems of toxicity and carbon source need to be solved so as to achieve high total nitrogen removal rate.

To reduce the negative impact of toxicity of PAH, activated sludge coupling with activated carbon adsorption, i.e. powdered activated carbon technology (PACT) was a promising method to remove aromatic compounds (Quan et al., 2005), such as phenol (Sundstrom et al., 1979; Cansado et al., 2012), 3,5-dichlorophenol (Widjaja et al., 2004), 2-chlorophenol (Aktas and Cecen, 2007). Particularly, it is also a cost-effective method due to its bioregenerability by the biomass bulk. Due to the low BOD5/N ratio of CGW, shortcut nitrification process seems feasible method, owing to less carbon resource demand as well as less oxygen demand. In the shortcut biological nitrogen removal (SBNR) process, ammonia is partially oxidized to nitrite and the nitrite is directly reduced to dinitrogen (N₂). It uses a shortcut microbial pathway instead of the complete oxidation via NO3–N in the overall reaction (Hwang et al., 2006). The process can be obtained through appropriate manipulation of pH, SRT, DO, free ammonia (FA) concentration (Yang et al., 2007). Besides, the NH3–N in CGW was in the range of approx. 100–200 mg/L, which ensured a successful start-up of short-cut biological nitrogen removal reactor (Park et al., 2010).

However, in previous studies of CGW wastewater treatment, work on improvement of total nitrogen removal was rare. No work on both eliminating toxicity and enhancement of denitrification via short-cut has been done in CGW wastewater treatment. So in the current study, performance of PACT coupling with SBNR was investigated. Moreover, the role of PACT in the TN removal was investigated. The process was tested to be feasible and preferable reference to further engineering application.

2. Methods

2.1. Experimental setup

Two quadrature aeration containers (shown in Fig. 1), one acted as PACT and the other one as SBNR reactor, were constructed of cubic plexiglass. The reactor unit with a working volume of 3.8 L was made of 16 cm in length, 16 cm in width, 15 cm in height. The horizontal flow sedimentation tank after PACT was of a total working volume of 0.7 L. The sludge from the sedimentation tank after PACT was of a total working volume of 0.5 L. The reactors were operated at room temperatures of 28 ± 5°C in summer (Yang et al., 2007; Wu et al., 2009), while in winter, the SBNR were maintained at 32 ± 1°C by electrothermal thread warming system.

2.2. Characteristic of sludge and wastewater

Seed sludge for PACT and SBNR was respectively obtained from the full-scale aerobic tank 1 (after anaerobic process) and aerobic tank 2 (after aerobic tank 1) treating Lurgi coal gasification wastewater at China Coal Longhua Harbin Coal Chemical Industry Co., Ltd. The plant had been operating over 2 years. The average temperature of the raw wastewater in 1 year was around 35°C. The sludge was grey-black with good settlement property. The PACT and SBNR tank were inoculated with around 20 and 15 g of volatile suspended. The VSS/SS ratio of the seed sludge was about 0.7.

The raw wastewater employed in the current study was synthesized according to the effluent composition of anaerobic reactors in the Lurgi gasification wastewater treatment plant (Coal Longhua Harbin Coal Chemical Industry Co., Ltd, China). The concentration of COD, BOD5, NH3–N, total phenols, volatile phenols in the real wastewater were 1350, 401, 154, 420 and 102 mg/L, respectively. NH3–N in the anaerobic outlet was slightly higher than that in the inlet (data not shown), indicating there was no NH3–N reduction but increase instead. And the BOD5/COD was approx. 0.30. The PACT tank was fed with the synthetic wastewater 1 (SW1) consisted of crude phenol obtained from the same company, phenol, sodium acetate, NH4Cl of analysis grade, and the following macro-nutrients: 50 mg/L MgSO4·7H2O, 20 mg/L K2HPO4, 20 mg/L CaCl2·2H2O, 15 mg/L FeSO4·7H2O, 10 mg/L KH2PO4, 15 mM/L NaHCO3. The main characteristic of SW1 was shown in Table 1.

During the start-up, the SBNR was fed with synthetic wastewater 2 (SW2), with phenol of 432 mg/L, NH4Cl of 550 mg/L (144 mg NH3–N/L) and NaHCO3 of 1728 mg/L (20.60 mM/L).

2.3. Start-up and operation

The two reactors were started separately. In the beginning, PACT tank was fed with raw wastewater. Gradually, the synthetic waster was added into the feed with ratio increasing to allow the biomass to acclimatize with SW1. With SRT controlled at 12–15 d, much NOB in the seed sludge could be washed and AOB and NO2 concentration started accumulated in the aerobic compartment of SBNR. Then steady-state operation lasted for months. 20 d and 15 d of SRT were used for PACT and SBNR respectively. When PACT was running stably and successfully, the followed SBNR was fed with PACT outlet. During this period, sodium acetate of 50 g/L as additional carbon resource was pumped into the SBNR anoxic compartment with flow rate of 0.01 L/h.
COD, BOD <sub>5</sub>, SS, VSS, total phenols (TP), oil, ammonia NH<sub>3</sub>–N and NO<sub>2</sub>–N were measured according to Standard Methods (APHA, 1998). The COD concentrations and TP in the PACT and NH<sub>3</sub>–N, NO<sub>2</sub>–N in the SBNR were analyzed once daily. The NO<sub>2</sub>–N and TN was analyzed once every 2 days. It was worth to note that for BOD<sub>3</sub> analysis, the sludge in the SBNR was used as the inoculum which was carried out twice, at 48 h intervals. Hybridization was followed by a thorough washing step, concentration for NSO190 and Ntspa662 were 55% and 35%, respectively. Hybridization buffer (0.9 mol/L NaCl, 20 Mmol/L Tris–hydrochloride pH 7.2) 0.01% sodium dodecyl sulfate, and formamide) and hybridization incubator at 46°C for 3 h. FISH probes used in the experiment were NSO190 with fluorescence tag FITC (for ammonia oxidizing β-Proteobacteria) and Ntspa662 (for Nitrosospira) with fluorescence tag TRITC. In the hybridization buffer, formamide concentration for NSO190 and Ntspa662 were 55% and 35%, respectively. Hybridization was followed by a thorough washing step, which was carried out twice, at 48°C for 20 min with 50 ml of pre-warmed washing Buffer. The slides were mounted with to avoid bleaching and examined with an OLYMPUSBX52 fluorescence microscope.

### Table 2
PACT operating condition.

<table>
<thead>
<tr>
<th>Condition</th>
<th>Inlet</th>
<th>PACT operation condition</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>SW2</td>
<td>Without PACT</td>
</tr>
<tr>
<td>2</td>
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</tr>
<tr>
<td>3</td>
<td>Effluent of PACT</td>
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</tr>
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<td>PAC dosage = 0.5 g/L, HRT = 12 h, DO = 2 mg/L</td>
</tr>
<tr>
<td>5</td>
<td>Effluent of PACT</td>
<td>PAC dosage = 1 g/L, HRT = 24 h, DO = 4 mg/L</td>
</tr>
</tbody>
</table>

### Table 3
Main characteristics of raw wastewater.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>COD (mg/L)</td>
<td>1207.4–1530.9</td>
</tr>
<tr>
<td>BOD&lt;sub&gt;3&lt;/sub&gt; (mg/L)</td>
<td>327.1–394.7</td>
</tr>
<tr>
<td>TOC (mg/L)</td>
<td>406.8–569.3</td>
</tr>
<tr>
<td>NH&lt;sub&gt;3&lt;/sub&gt;–N (mg/L)</td>
<td>149.6–159.3</td>
</tr>
<tr>
<td>Volatile phenols (mg/L)</td>
<td>459.2–608.0</td>
</tr>
<tr>
<td>Bicarbonate alkalinity (mmol/L)</td>
<td>10–15</td>
</tr>
<tr>
<td>pH</td>
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</tr>
</tbody>
</table>

### 2.4. Effect of PACT performance on total nitrogen removal in the SBNR

In order to investigate the relationship between PACT and SBNR, nitrite accumulation and TN removal efficiency by SBNR was compared in different scenarios- with PACT under different operating conditions, i.e. with different HRT, DO and powdered activated carbon (PAC) dosage (shown in Table 2). Each lasted more than 20 days.

### 2.5. Enhancement of biodegradability by PACT

The biodegradability test was conducted to study the role of PAC in nitrogen removal. SW1 and PACT outlet under condition 5 was directly used as test objects and SW1 after 12 h adsorption by 1 g/L PAC at DO of 4 mg/L, i.e. the supernatant after adsorption by activated carbon (SAAC) was selected as control. Gas chromatography–mass spectrometry (GC–MS) was assayed for the three samples above to valuate the PAC adsorptive preference. Thereafter, FTIR test was used to highlight and validate the GC–MS analysis.

### 2.6. Fish

Sludge from PACT and SBNR were fixed in 4% paraformaldehyde solution from 8 h at 4 °C on day 30. After dispersed by ultrasonic treatment for 1 min, 5 μL of the sample was placed on a slide coated with 0.1% gelatin and dried overnight. Then the specimen was immersed and dehydrated by successive 50%, 80%, and 99.5% ethanol solution (3 min each) and air dried. Then after 90°C for 3 h. FISH probes used in the experiments were NSO190 with fluorescence tag FITC (for ammonia oxidizing β-Proteobacteria) and Ntspa662 (for Nitrosospira) with fluorescence tag TRITC. In the hybridization buffer, formamide concentration for NSO190 and Ntspa662 were 55% and 35%, respectively. Hybridization was followed by a thorough washing step, which was carried out twice, at 48°C for 20 min with 50 ml of pre-warmed washing Buffer. The slides were mounted with to avoid bleaching and examined with an OLYMPUSBX52 fluorescence microscope.

### 2.7. Analytical methods

COD, BOD<sub>5</sub>, SS, VSS, total phenols (TP), oil, ammonia NH<sub>3</sub>–N and NO<sub>2</sub>–N were measured according to Standard Methods (APHA, 1998). The COD concentrations and TP in the PACT and NH<sub>3</sub>–N, NO<sub>2</sub>–N in the SBNR were analyzed once daily. The NO<sub>2</sub>–N and TN was analyzed once every 2 days. It was worth to note that for BOD<sub>3</sub> analysis, the sludge in the SBNR was used as the inoculum which was carried out twice, at 48 h intervals. Hybridization was followed by a thorough washing step, concentration for NSO190 and Ntspa662 were 55% and 35%, respectively. Hybridization was followed by a thorough washing step, which was carried out twice, at 48°C for 20 min with 50 ml of pre-warmed washing Buffer. The slides were mounted with to avoid bleaching and examined with an OLYMPUSBX52 fluorescence microscope.

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### 3. Result and discussion

#### 3.1. SBNR start-up and steady operation

##### 3.1.1. Nitrite accumulation and TN removal

For the SBNR in this study, we used a similar strategy for suspended-growth short-cut biological nitrogen removal reported by Park et al. (2010). The SBNR was started up quickly and then stably operated even increased the NH<sub>3</sub>–N concentration in the inlet. After 23 days, the nitrite accumulation rate in the aerobic compartment reached a very high level (~1.09 mg NO<sub>2</sub>–N/(gVSS h)). The conversion rate of NH<sub>3</sub>–N to NO<sub>2</sub>–N in the aerobic compartment was very high to 95.15% (shown in Table 3), which indicated an absolute superiority of AOB to NOB. The anoxic compartment exhibited a high TN removal efficiency of 89.78%, especially with phenol as carbon source. The microscopy of the aggregated biomass fraction from the aerobic compartment explained a dominance of cocoid and regularly shaped cells arranged in tight clusters. FISH assay revealed that most of these cells reacted with the probes specific for the beta-proteobacterial AOB, belonged to the genus Nitrosomonas. The probes Ntspa662, which are specific for the Nitrosospira spp, showed very weak signal.

##### 3.2. Influence of PACT performance on SBNR

Figs. 2 and 3 present the relationship between PACT and SBNR performance. As stated above, SBNR achieved high short-cut nitrogen removal efficiency if feeding with SW2. However, when feeding with raw wastewater (condition 2, PACT canceled), the concentration of nitrite decreased dramatically from 137.01 ± 14.15 mg/L to 26.37 ± 4.08 mg/L. The conversion rate of NH<sub>3</sub>–N to NO<sub>2</sub>–N was only 18.88%, which indicated a severe suppression of AOB activity by the raw water. Thereafter, SBNR was recovered in more than 15 days (data not shown in Fig 2). Converting to condition 3, PACT removed only 17.20% COD and 22.27% of total phenol with 0.2 mg/L PAC dosage, 4 h HRT, 0.5 mg/L DO. Correspondingly, the SBNR was not improved much. The concentration of NH<sub>3</sub>–N was still at high level. After further increasing PAC dosage and DO, the PACT achieved higher COD and phenol removal efficiency in condition 4, up to 61.3% and 68%, respectively. Accordingly, the NH<sub>3</sub>–N to NO<sub>2</sub>–N conversion rate and TN removal rate in SBNR was increased to 60.73% and 48.34%, respectively. Besides, when PACT performance was further improved under optimized condition 5, removal efficiency for COD and phenol were 85.80% and 90.30%, respectively. Thus the less harmful outlet set good foundation for short-cut removal process. And it was reasonable to obtain NH<sub>3</sub>–N to NO<sub>2</sub>–N conversion rate of 86.89% in SBNR aerobic compartment. Apparently, a sharp increase in NO<sub>2</sub>–N also
occurred but in fact it did not come from the inlet but produced by nitrite-oxidizing bacteria in PACT. The TIC/TNH (total inorganic carbon: total nitrogen ammonia) of SBNR inlet was 71.76/79.5, i.e. 0.90 and BOD5/TNH was 335/79.5, i.e. 4.21. At this value, the reduction in conversion rate was in the range concluded by Ganigué et al. (2012). So compared with condition 1, the slight restrain on AOB might be partly attributed to the CO2 produced during oxidation of organic matter. It acidified the wastewater and reduced the buffering capacity to balance protons produced (see bicarbonate alkalinity variation in Table 3) during TNH oxidation and eventually reduced NH3–N conversion rate (Vilar et al., 2010). In the anoxic compartment, the denitrification bacteria can utilize the remaining BOD5 almost completely, resulting in a TN removal efficiency of 75.54%. Under the optimal Condition 5, PACT exhibited removal percentages of 85.80%, 83.83%, 36.88%, 90.30%, 64.00% for COD, BOD5, NH3–N, TP, oil, respectively (shown in Table 4). It was also necessary to note that the chromaticity decreased dramatically after PACT process, which is also in good agreement with Mezohegyi et al. (2012).

It can be concluded that SBNR performance-in terms of nitrite accumulation and TN nitrogen removal rate was improved by PACT performance. The operation parameters for PACT, among which PAC dosage was one of the most important (proved by orthogonal experiment, not shown), influenced much on the pollutants removal and therefore influenced much on the nitrogen removal efficiency.

### 3.3. Biodegradability improvement by PACT

From the above findings, it could be concluded that PACT reactor was indispensible for TN removal of the combined system. And according to the orthogonal test result (not shown), PAC dosage was the most significant among the four important factors. To explore the role of PAC, biodegradability tests compared SW1, outlet of PACT and SAAC. BOD5/COD was tested as 0.29 ± 0.01, 0.32 ± 0.02 and 0.34 ± 0.04, respectively. So both the sludge and PAC present in PACT increased the biodegradability. Further analysis can be made based on the organic composition of the 3 streams above (shown in Table 5). The phenol with double or multiple methyl groups and other refractory compounds were partly or completely eliminated by PAC. Specifically, among these compounds, the indole, 6-methyl-4-indanol, N-phenyl propanamide, 4,4-dimethyl-N-phenyl hexanamide, defined as nitrogen heterocyclic compounds (NHCs) and have been proved to cause great toxicity and harm to the sludge activity, all carried the chromophore, such as –N@, –CO–NH, etc. This fact corresponded with the significant decrease in chromaticity. Thus, the PAC present in the PACT protected the suspended biomass from suffering, while the biomass degraded the remaining phenols, long-chain or aromatic hydrocarbons (Liu et al., 2012), some of which belonged among recalcitrant substances, such as 5,5-dimethyl-2, 4-imidazolidinedione. Sher et al. (2000) found that PACT sludge with high sludge ages was much more resistant to toxic exposure with changes in the biomass rather than sorption on carbon. And the activated sludge also had large and resilient adsorption capacity. It was also revealed that PAC preferentially absorbed aromatic compounds with complex molecule structure rather than simple organic acid, the intermediates produced from the anaerobic metabolism, such as butanoic acid, pentanoic acid, propanoic acid (data not shown). These simple organic acids were quite beneficial for the biomass. Not only were they easily degraded but they might also play a role of co-substrate to promote the degradation of the remaining recalcitrant substances (Bajaj et al., 2008; Ghevariya et al., 2011). Therefore, PACT integrated the advantages from two aspects: adsorption of recalcitrant compounds by activated carbon; adsorption selectivity was also helpful for the sludge to uptake the remaining refractory compounds.

### Table 3
Analysis of N conversion and bicarbonate alkalinity under condition 1–5.

<table>
<thead>
<tr>
<th>Condition</th>
<th>NH3–N to NO2–N conversion rate (%)</th>
<th>TN removal rate (%)</th>
<th>Bicarbonate alkalinity (mM/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>95.15</td>
<td>89.78</td>
<td>0.50 ± 0.16</td>
</tr>
<tr>
<td>2</td>
<td>18.88</td>
<td>17.68</td>
<td>1.27 ± 0.16</td>
</tr>
<tr>
<td>3</td>
<td>36.06</td>
<td>24.99</td>
<td>0.91 ± 0.04</td>
</tr>
<tr>
<td>4</td>
<td>60.73</td>
<td>48.34</td>
<td>0.42 ± 0.05</td>
</tr>
<tr>
<td>5</td>
<td>86.89</td>
<td>75.54</td>
<td>0.11 ± 0.07</td>
</tr>
</tbody>
</table>

![Fig. 2. COD and phenol removal efficiency of PACT under conditions 1–5.](image-url)
3.4. Adsorptive preference of PAC for CGW

FTIR spectral analysis allows the identification of different functional groups of the organic substance present in the wastewater. By making a contrast in FTIR spectra of raw wastewater and SAAC, the adsorptive preference of PAC can be concluded. In the FTIR spectrum, the complex water composition led to many peaks in a wide range of spectrum. As is shown, these two streams had much in common in terms of trends and peaks: The strong peaks near 3400 cm\(^{-1}\) could be due to the hydroxyl group \(\text{v}(\text{O–H})\), the \(\text{v}(\text{C–H})\) and also \(\text{v}(\text{N–H})\) stretches; peaks around 2900–3000 cm\(^{-1}\) indicated the presence of \(\text{v}(-\text{CH}_3)\); the strong peaks near 1000–1300 cm\(^{-1}\)
was typical of $\nu(-\text{NH})$ and C–O which could be attributed to the presence of nitrogen groups and phenol, ester or carboxyl groups (COOH). However, these two samples also presented distinction in intensity at some wavelengths. For example, the raw wastewater exhibited an obvious set of peaks emerging in the region of 1600–1700 cm$^{-1}$, which could be related with $\nu(-\text{NO}_2)$, and $\nu(C=O)$ associated with $\nu(-\text{CONH})$ i.e. amino. These were just common chromophoric (Bansal and Sud, 2012) and auxochrome group. In the lower region of the spectra, the raw wastewater showed the more intense absorption bands near 680–880 cm$^{-1}$, which could be out-of-plane O–H bending attributed to the presence of substituted aromatic. In contrast, these bands are less noticeable on the FTIR spectra of SAAC which contains less aromatic hydrocarbons. Therefore, agreeing with GCMS result, FTIR spectra indicated that PAC adsorbed some organic compounds, leading to a decrease in chromaticity observed.

4. Conclusions

The following conclusion can be drawn from this study:

The developed PACT and SBNR combined process was investigated to enhance the total nitrogen (TN) removal efficiency for the UASB effluent treating CGW. The SBNR performance was highly and positively related with PACT removal efficiency. The PAC partly or completely removed recalcitrant nitrogen heterocyclic compounds (NHCs), thus allowing SBNR to be loaded with a relatively harmless and higher BOD$_5$/COD influent. The study demonstrated that the PACT system could serve as a technically feasible method as a pretreatment for short-cut nitrogen removal for coal gasification wastewater.

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