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Analysis of Evolved Species and Estimation of the Combustion Emissions of PACB and AAC Preservatives Using TGA–IR and CEM Techniques

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Alkyl Ammonium Compound (AAC) and Protein Ammonia Copper Borate (PACB) are two types of waterborne preservatives. In this paper they were examined, first by using thermogravimetric analysis (TGA) under different heating rates (5 or 40 ˚C/min), second by using thermogravimetric analysis with infrared spectrometry (TGA–IR), and third by using continuous emission monitoring (CEM) techniques. The results obtained from the TGA tests found that the decomposition temperature of AAC and PACB at the heating rate of 5 ˚C/min was lower than that at 40 ˚C/min. Regardless of the different heating rates, the decomposition temperatures of PACB were all higher than those for AAC, and the char (wt %) of the PACB (65.38 wt % for 5 ˚C/min and 66.92 wt % for 40 ˚C/min) was more than that of the AAC (0.042 wt % for 5 ˚C/min and 0.046 wt % for 40 ˚C/min). The IR response of AAC and PACB in the TGA–IR tests showed that at a heating rate of 5 ˚C/min, the evolved species of AAC were less than those of PACB. However, at a heating rate of 40 ˚C/min the numbers of evolved species of AAC were much higher than those of PACB. Considering the decomposition step (thermal time or temperature) at different heating rates in the TGA tests and the IR response from the TGA–IR analysis, the results showed that at the slow–heating regime, the concentrations of CO_2 and the evolved species of $=CH_2$ groups produced by PACB were higher than those of AAC, but at 40 ˚C/min the results were reversed. The results of combustion emissions obtained by CEM techniques found that the emission gas temperature of AAC was higher than that of PACB. Both O_2 and CO_2 reached a plateau curve until the end of combustion, during which time they were closely related. The highest emission quantity of CO was about 94.0 ppm for AAC, but it was 120.0 ppm for PACB. The emission quantity of SO_2 for each type of sample was zero. The maximum NOx for AAC was about 49.5 ppm, but it was insignificant for the amount of PACB during combustion. The above results of the TGA–IR and CEM analysis allowed us to determine the kinds of evolved species and the emission gas concentrations of O_2 and CO_2, the emission contents (CO, SO_2, NOx), and the temperature of the emission gases for referencing the thermo–decomposition processes of AAC and PACB during combustion.

Keywords: Alkyl Ammonium Compound (AAC), Protein Ammonia Copper Borate (PACB), Char, Evolved Species, Combustion Emissions

INTRODUCTION

Wood preservatives have been used to preserve wood from insects, fungi and water damage for the past few decades. This is due to the fact that preservatives provide an extra layer of protection for any wood product when used for any outdoor application. Waterborne (water–based) preservatives are often used when cleanliness, printability and objectionable odor of the treated woods are required. These preservatives include chromated copper arsenate (CCA), ammoniacal copper quats (ACQ), and others. Other preservatives in CNS 14495 O 1048 include alkyl ammonium compound (AAC), ammoniacal copper azole (CuAz), inorganic boron (borax/boric acid), etc. Boron preservatives are very effective against decay, termites, beetles, and carpenter ants (Ibach, 1999), but they provide little resistance to leaching after the wooden has been used above ground where it is protected from wetting for a longer period of time. One of the more common formulations used in Taiwan contains mainly copper and borate, and is known as protein ammonia copper borate (PACB) because protein, especially the feather protein used, has shown high resistance to leaching and has proven to be a very good performer for wood and bamboo products (Fan, 2003; Mazela and Izabela, 2003; Chang *et al*., 2004).

At present much attention is being given to the disposal of wood preservatives, because during combustion they emit toxic and corrosive gases (Cox, 1991; Cooper *et al*., 2003; Solo–Gabriele *et al*., 2003). Exposure to these toxic emissions is harmful to humans and in addition can cause damage to expensive equipment (Ladomersky, 2000; Kercher and Nagle, 2001). With the use of thermogravimetric analysis with infrared spectrometry (TGA–IR), it is possible to simultaneously monitor any evolved spe-

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cies, the decomposition temperature and the concentrations formed (absorbance), as well as the evolution profile of certain compounds. IR spectrometry is a common method for evaluating a functional base when a material undergoes degradation (Lin *et al*., 2004; Lin and Murase, 2007). At the same time, Continuous Emission Monitoring (CEM) techniques are widely utilized for detecting gaseous emissions such as CO, SO_2, NOx, O_2 and CO_2 concentrations at the chimney exit of furnaces to determine the concentrations of various pollutants (Jang, 2000).

In our earlier works (Lin *et al*., 2007; Lin and Murase, 2007) we established some referable results for CCA and ACQ. One result was the emissions during combustion and the char after combustion. Another result was the use of TGA in combination with TGA–IR for analyzing the thermal behavior of 3 types of preservatives, which led to useful results, including the identification of pyrolysis products. To elucidate the comprehension of the thermal properties of AAC and PACB, a comparison was made between the slow–heating regime and the fast–heating regime in an air atmosphere. The examinations were divided into three categories. The first category involved the study of thermal decomposition, and examined the effectiveness of the thermal properties of AAC and PACB using thermogravimetric analysis (TGA). In the second category, TGA–IR was applied to monitor the effectiveness, on an analytical scale, of the two water–based preservatives and to examine the emissions of certain compounds in each particular analysis. The third category was applying CEM to analyze the combustion emissions during the combustion of both preservatives. The goal of this research was to acquire fundamental knowledge of the pyrolysis temperature (obtained from TGA), and the decomposition time of the evolved species from the IR response (obtained from TGA–IR) of AAC and PACB, while at the same time obtaining an air pollution (obtained from CEM) reference for gas emissions. The obtained results were also compared with the results of CCA and ACQ from previous works (Lin *et al*., 2007; Lin and Murase, 2007).

MATERIALS AND METHODS

Wood preservatives

The basic properties of both commercial wood preservatives, provided by Te Feng Lumber Co. Ltd., Taiwan, are as follows:

a. Alkyl Ammonium Compound (AAC): This waterborne (water–based) preservative is light yellow in color, and its main content is 35% didecyl dimethyl ammonium chloride (DDAC). The solid content is about 86.50 (0.001) %, and its specific gravity is 0.890 (0.001) at 27.0 °C, with a pH of 7.11.

b. Protein Ammonia Copper Borate (PACB): PACB is deep blue in color, and basically consists of 43 g of protein with, 200 mL ammonia. The basic weight rate (%) of copper to boron was 32 g of copper to 8 g boron with 996 mL water. Its effective content was 12.8 (kg/cm^3) (Chang *et al*., 2004). The solid content was about 0.35 (0.01) %, and its specific gravity was 1.007 (0.001) at 25.0 °C, with a pH of 9.78.

Thermogravimetric analysis (TGA)

In this experiment, TGA was carried out using a Perkin–Elmer TGA 1, fully supported by computer–controlled software options from Perkin–Elmer Thermal Analysis Systems for control and data handling. The sampling and the analyzed conditions, including the air flow rate and two kinds of heating rate for the TGA tests were the same as in previous reports (Lin *et al*., 2006; Lin and Murase, 2007), except that the heating range was between 50 to 800 °C while the measurements were carried out.

Thermogravimetric analysis with infrared spectrometry (TGA–IR)

This analysis was carried out using a TGA (Perkin–Elmer Pyris 7 TGA model), linked to a secondary method of analysis, the Fourier–Transform Infrared Spectroscopy (using a Perkin–Elmer FTIR), which was able to identify the evolved species during the TGA measurements (Statheropoulos and Kyriakou, 2000). The TGA and FTIR (TGA–IR) were connected using a transfer line with a continuous scanner in a gaseous cell. The infrared spectrometric (IR) response data was recorded by a personal computer. The spectrum time–based version 2.0 software (2000, Perkin–Elmer, Inc.) obtained and analyzed the evolved species as they occurred. The sampling and the calculation of the IR response obtained by the spectrum version 5.3 (2005, Perkin–Elmer, Inc.) were the same as for the previous report (Lin and Murase, 2007), and the conditions for heating and air flow rates were set the same as for the Perkin–Elmer TGA 1, described above. The evolved species analysis was performed for two kinds of water–based preservatives.

Experimental combustion methods

A Flue–gas Analyzer (MSI 2000, W. Germany) was used to examine the gas emitted from a flammability test cabinet (with reference to CNS 7614 A3125, NCYU handmade) during the combustion of AAC and PACB. The experimental design and tested methods for the CEM techniques were the same as for the previous reports (Lin *et al*., 2001; Lin and Huang, 2004; Lin, 2005; Lin *et al*., 2007). Six replicates of each sample of the preservatives, after first having been oven–dried, were placed individually into a crucible on a clay triangle with a triangular stand. The first cycle run in the flue–gas analyzer did not include combustion with a sample in the test cabinet to be certain that the measured value of various emission gases was zero, and that the gas temperature was at room temperature. The sample was ignited and then examined for 9 cycles continuously. The CO, SO_2 and NOx gases were measured in parts per million (ppm). The O_2 and CO_2 concentrations were defined as the volume percent (VOL %) of the air in the emission exit. The gas temperature was measured at the same time.

RESULTS AND DISCUSSION

Thermal decomposition of AAC and PACB

To comprehend the thermal properties of the AAC and PACB, the preservatives samples, after having been oven–dried, were measured by TGA. Decomposition profiles were obtained while the samples were being heated at a rate of 5 or 40 ˚C/min in air, between 50 ˚C (initial temperature) and 800 ˚C (end temperature). The relationships between the TGA and DTG curves, the temperature and weight loss, and the derivative weight for AAC and PACB are shown in Fig. 1.

Regardless of the heating rate, both water–based preservatives typically showed a gradual weight loss. The thermogram, resulting from the analysis of AAC at a heating rate of 5 ˚C/min (solid line) and 40 ˚C/min (dash line), respectively, showed that there was a subtle change (shoulder) at temperatures between 50 and about 250 ˚C. The onset of weight loss under air was observed at a temperature of 233.42 ˚C for the heating rate of 5 ˚C/min, and at a temperature of 282.09 ˚C for the heating rate of 40 ˚C/min. These findings are supported by the derivative (DTG) curves, which show the peak at a temperature of 236.37 ˚C for 5 ˚C/min, and at a temperature of 287.52 ˚C for 40 ˚C/min (see bottom of Fig. 1). The amount of char for both heating rates, at a temperature of 800 ˚C, 5 ˚C/min was 0.042 wt % and for 40 ˚C/min it was 0.046 wt %.

The TGA results of PACB for both heating rate are also shown in Fig. 1. The weight loss of PACB at a heating rate of either 5 ˚C/min (dotted line) and 40 ˚C/min (short dash line) showed that there were five shoulders for each heating rate, and the weight loss decreased gradually. The onsets of weight loss for the 40 ˚C/min heating rate were observed at temperatures of 122.03, 253.32, 324.89, 493.52 and 697.17 ˚C, respectively. For the heating rate of 5 ˚C/min, the weight loss was obvious at temperatures of 98.60, 224.01, 314.75, 468.24 and 605.11 ˚C, respectively. These findings are supported by the DTG curves of PACB (see bottom of Fig. 1). For PACB, the amount of char at a temperature of 800 ˚C, was 65.38 wt % at 5 ˚C/min and 66.92 wt % at 40 ˚C/min. These numbers are higher than those for AAC at either heating rate.

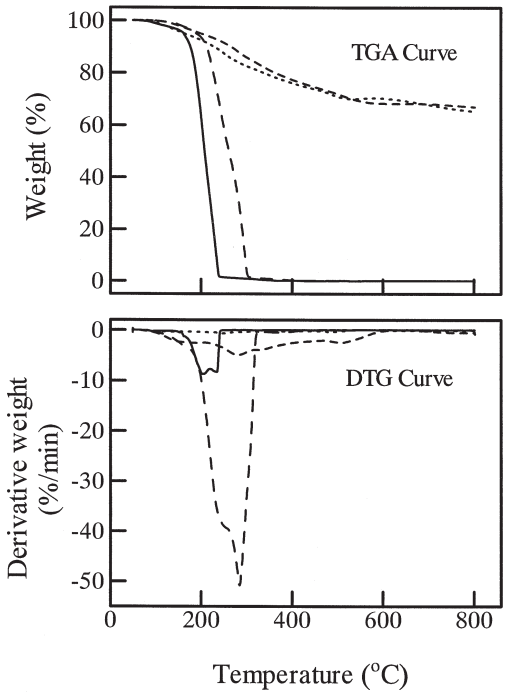

Fig. 1. TGA and DTG curves for 2 types of wood preservatives (AAC and PACB) in air at 5 or 40 ˚C/min.
Symbols: ——: AAC (5 ˚C); – – – : AAC (40 ˚C); ·····: PACB (5 ˚C); - - - -: PACB (40 ˚C).
Notes: AAC: Alkyl Ammonium Compound; PACB: Protein Ammonia Copper Borate; 5 ˚C: 5 ˚C /min of heating rate; 40 ˚C: 40 ˚C/min of heating rate.

The above results showed that the decomposition temperatures of AAC and PACB for a heating rate of 5 ˚C/min were lower than those at 40 ˚C/min. Regardless of the heating rate, the decomposition temperatures of PACB were all higher than for AAC. This indicated that the thermal decomposition of AAC and PACB was influenced by the heating regimes in an air atmosphere. Compared to the char results of CCA and ACQ (Lin and Murase, 2007), the char of PACB was higher than that of ACQ–1 and lower than that of CCA. This is because PACB is a copper–based wood preservative (Ibach, 1999), and its main chemical composition includes protein, ammonia, copper and borate. Moreover, some researches (Hirata *et al.*, 1993; Kercher and Nagle, 2001; Cooper *et al.*, 2003; Helsen and Bulck, 2005; Lee *et al.*, 2005) reported finding large amounts of Cu (copper) in the char of copper–based wood preservatives, such as CCA. Even for ACQ preservatives, a combination of copper and organic biocides, the char left a high amount of inorganic metal elements, Cu, of about 50.14%. It is evident that the Cu from the preservative (PACB) remains after combustion.

Infrared spectrometric response of AAC and PACB

Fig. 2 shows the infrared spectrometric (IR) response of the AAC and PACB for the heating rate of 5 ˚C/min (on the left side of Fig. 2) and the heating rate of 40 ˚C/min (on the right side of Fig. 2) in the TGA–IR measurements. The changes in absorbance (concentration of evolved species, %) correspond to the increase in decomposition time. For the IR response of AAC at a heating rate of 5 ˚C/min it was increased to about 1363.03 sec (112.76 ˚C) from the start of decomposition. Subsequently, this tendency reduced to about 5088.24 sec (423.19 ˚C). After that, the rate of change in concentration of evolved species increased until the end of decomposition. The IR response of PACB at a heating rate of 5 ˚C/min rose linearly to about 5759.29 sec (479.11 ˚C). Subsequently, this tendency reduced to about 6777.74 sec (563.98 ˚C). After that, the high point of absorbance occurred at the end of the decomposition. This suggests that at a heating rate of 5 ˚C/min, the concentration of evolved species for PACB is higher than that for AAC.

The right side of Fig. 2 also shows the IR response of the AAC and PACB at the heating rate of 40 ˚C/min in the TGA–IR measurements. The changes in concentra-

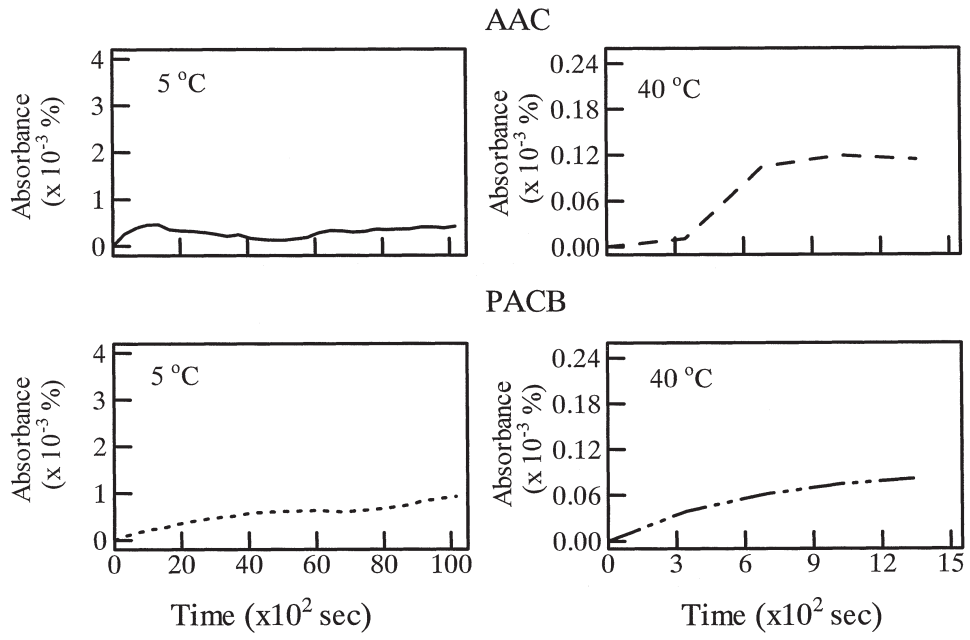

Fig. 2. IR response of 2 types of wood preservatives (AAC and PACB) in air at 5 or 40 °C /min from TGA–IR analysis.
Symbols: The symbols are the same as Figure 1.
Note: Abbreviations of AAC, PACB and 5 °C, 40 °C are the same as Figure 1.

tion of evolved species also correspond to the increase in decomposition time for the two preservatives. For the AAC, it slowly increased to about 344.08 sec (222.72 °C) from the start of decomposition. Subsequently, the increase in decomposition rose linearly to about 681.83 sec (447.88 °C), and then leveled off until the end of decomposition. The change in absorbance for PACB slowly increased until the end of decomposition. Comparing the IR response of both preservatives at both heating rates, it was found that the absorbance levels at 5 °C/min for PACB were higher than those for AAC. However, at 40 °C/min the results were reversed. This indicates that for wood preservatives the concentration of evolved species is related to the heating rate and the chemical composition, because of the variation in decomposition steps (Fig. 1).

TGA–IR spectra of AAC and PACB

The evolution of decomposition time (or temperature) on absorbance and wavenumbers (basically the IR spectra) for AAC and PACB is shown in Fig. 3. The distribution of wavenumbers at the end of each 600 sec period for the heating rate of 5 °C/min (the left side of Fig. 3) and at the end of each 100 sec period for 40 °C/min (the right side of Fig. 3), during decomposition, was analyzed.

The TGA–IR spectra of AAC and PACB at a heating rate of 5 °C/min are shown in the left side of Fig. 3. For the AAC, the range from 1800 sec (at a temperature of about 149 °C in the TGA tests) to the end showed a strong peak at 2934 cm^{-1}, which was attributed to $=CH_2$ groups with a symmetric stretching vibration. The carbon dioxide (CO_2) peak, located at 2296 cm^{-1}, showed up around 3600 sec (349 °C) and remained steady to the end. Another CO_2 weak peak was located at 669 cm^{-1} and remained there from about 2400 sec (249 °C) to the end. In addition, some of the weak peaks at 3791, 1744, 1278, 1100 and 732 cm^{-1} were due to the N–H stretching of NH_2 moieties, C=O (carbonyl group) stretching, the group of $N–NO_2$ stretching, C–O stretching and –OH out–of plane wagging from about 1200 sec (149 °C) to the end. For PACB, the stronger peaks, at 3752, 3620, 1714, and 1574 cm^{-1}, were due to the N–H stretching of NH_2 moieties, the OH group, C=O stretching and the group of C–N stretching, all ranging from the beginning of the combustion to the end. These numbers all showed an increasing trend, especially the N–H stretching of NH2 moieties. Except for the evolved species, several weak peaks at 1256, 1124, 998, 746 and 603 cm^{-1} emerged due to $N–NO_2$, C–O, OH groups, $=CH_2$ and CO_2 during the period of this TGA–IR test. The above results indicate that for the slow–heating regime (5 °C/min), the evolved species of AAC from the TGA–IR response were less than those of PACB.

For the heating rate of 40 °C/min, the TGA–IR spectra of AAC and PACB are shown in the right side of Fig. 3. For AAC, the peaks at 2945 and 2307 cm^{-1} were due to the $=CH_2$ groups with a symmetric stretching vibration and the CO_2 peak from about 300 sec (243 °C) to the end. A higher concentration of $=CH_2$ groups and CO_2 peak was seen between 400 and 1100 sec (310 to 777 °C). In addition, the absorbance from 400 sec to the end (260 to 799 °C) showed slight peaks at 1744, 1278, 990 and 732 cm^{-1}, which were attributed to C=O stretching, $N–NO_2$, OH group and $=CH_2$. For PACB, the absorbance peaks of CO_2, C–O, –OH and $=CH_2$ groups at 2291, 1078, 1026 and 814 cm^{-1} showed from about 400 sec (310 °C) to the end. In addition, this time period and the decom-

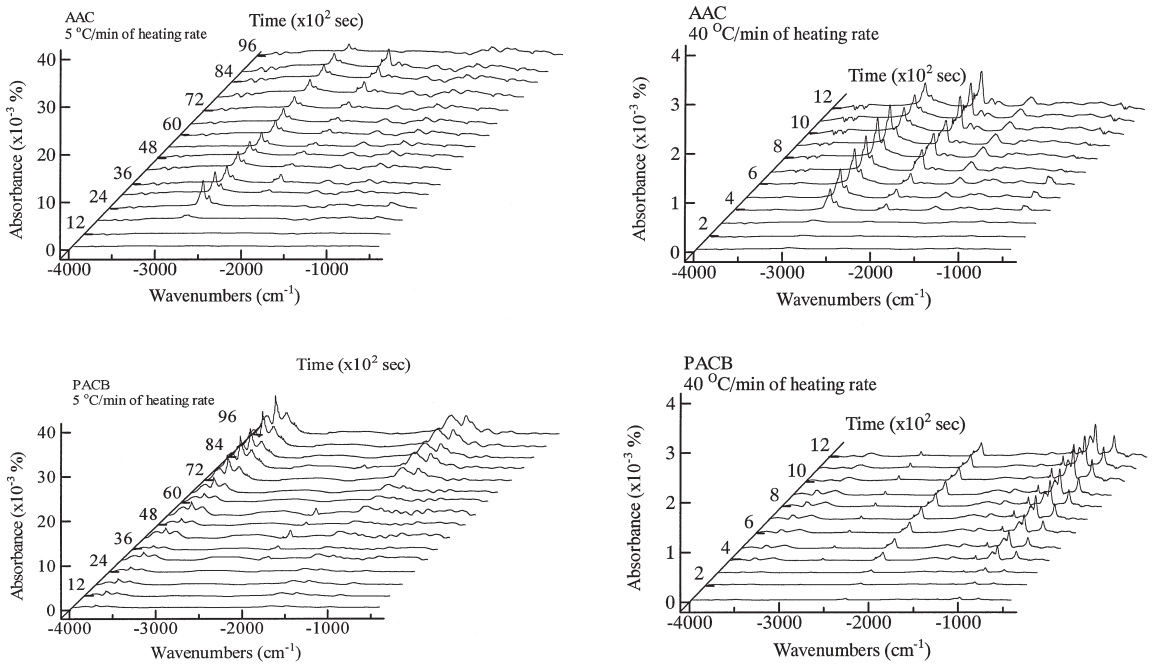

Fig. 3. TGA–IR spectra of 2 types of wood preservatives (AAC and PACB) in air with 5 or 40 °C/min.
Note: Abbreviations of AAC and PACB are the same as Figure 1.

position temperature to the end also showed six slight peaks of absorbance at 3730, 3521, 2972, 1514, 1267 and 617 cm^{-1}, which were attributed to N–H, –OH, $=CH_2$, C–N, $N–NO_2$ groups and CO_2 etc. These results indicate that at a heating rate of 40 °C/min, AAC presented a much larger amount of evolved species than PACB.

When comparing the results of the IR response with the TGA–IR spectra of CCA and ACQ (Lin and Murase, 2007), we found that the concentration of the evolved species of both AAC and PACB was much higher than that of CCA, but less than that of ACQ. This is because these preservatives are composed of different chemical compounds that emit different volatiles during decomposition.

Emission gases from AAC and PACB

The results between the emission gas temperatures, concentration of O_2 and CO_2, and the combustion times for AAC and PACB are shown in Fig. 4. The increase in emission gas temperature corresponded to the increase in combustion time. The emission gas temperature for AAC increased linearly to about 129 °C from the start of the combustion at 113.5 sec and then decreased linearly to about 106 °C at 151.0 sec. Then it finally decreased slightly to the end at 101 °C. However, for PACB it was increased steadily to the end with the temperature at about 100 °C. The emission gas temperature for AAC was found to be higher than that of PACB. This indicates that the ignition point and/or the heat decomposition temperatures of both preservatives are different because of the different chemical compounds (Lin *et al.*, 2001; Lin *et al.*, 2004; Lin *et al.*, 2007).

The results also showed that an increase of CO_2 and a decrease of O_2 corresponded to the increase in com-

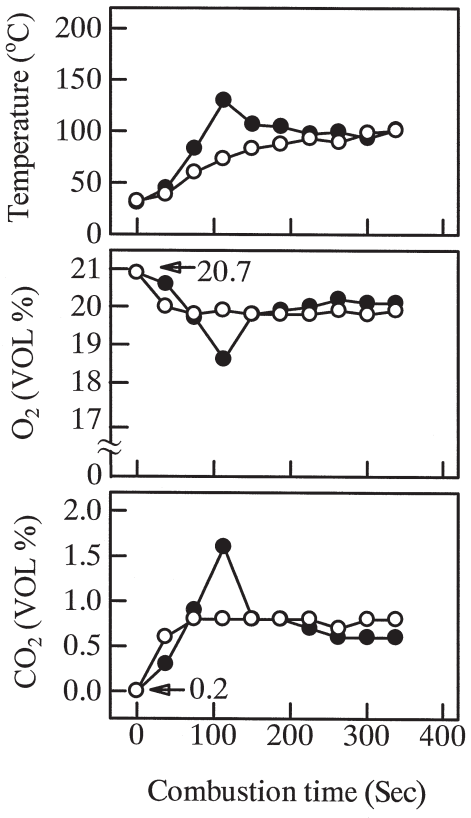

Fig. 4. Relationships between combustion time and emission gas temperature, O_2, CO_2 concentration for AAC and PACB.
Symbols: —●—: AAC; —○—: PACB.
Note: Abbreviations of AAC and PACB are the same as Figure 1.

bustion time for AAC and PACB, as shown in the middle and bottom of Fig. 4 respectively. The lowest % O_2 for AAC was about 18.6%, and the peak for CO_2 was about 1.6%. The gas temperature reached about 105 to 110 ˚C after 150.0 sec and remained stable until the end of the combustion period. For PACB, the concentration of O_2 decreased linearly from 20.7% to about 20.0%. On the other hand, the concentration of CO_2 increased from 0.2% to about 0.7%. Both O_2 and CO_2 then reached a plateau until the end of the combustion time. The concentration of O_2 was negatively related and CO_2 was positively related to the emission gas temperature. The concentration of O_2 and CO_2 were closely related to each other. These results agree with those of Lin *et al.*, 2001; Lin and Huang, 2004; Lin, 2005; Lin *et al.*, 2005; Lin *et al.*, 2007.

The results of the emission contents for the two types of preservatives are shown in Fig. 5. The peak for CO gas for AAC was found to be at 113.5 sec with 94.0 ppm after the beginning of combustion, and then decreased until the end of the combustion period. For PACB a maximum value of CO was reached at about 75.0 sec with 120.0 ppm from the start of combustion and then it decreased until the end. This indicated that the emission of CO gas is not only related to the ignition time (Ladomersky, 2000; Lin *et al.*, 2001; Lin *et al.*, 2004; Lin *et al.*, 2007), but is also related to the incomplete combustion at different combustion times due to the different chemical compounds. This experiment found that no SO_2 was being emitted by any of the preservatives during combustion. The results (see the bottom of Fig. 3) also showed that both AAC and PACB produced NOx gas. Although the amount of NOx gas produced was insignificant for PACB during the combustion, the peak of NOx gas for AAC was about 49.5 ppm at about 113.5 sec of the combustion period. NOx is considered to be the main source of pollution from burning AAC, and its amount was higher than that of ACQ and CCA (Lin et al., 2007). It is inferred that the ignition point and/or the heat decomposition of the preservatives may influence the combustion time and the quantity of emission gases, including NOx. The reason for NOx gases being created through the burning of these preservatives is due to the fact that they include N in their chemical compounds (Kercher and Nagal, 2001; Lin *et al.*, 2006 and 2007).

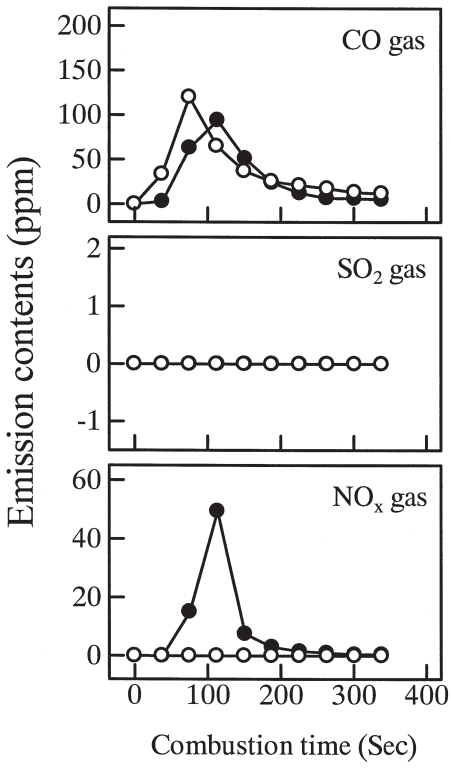

Fig. 5. Relationships between combustion time and emission contents for AAC and PACB. Note: Abbreviations of AAC, PACB and symbols are the same as Figure 1 and 4.

CONCLUSIONS

This paper examined the pyrolysis temperature, the decomposition time of the evolved species, and the emission gas concentration of AAC and PACB. The results were then compared with those of previous works (Lin *et al.*, 2007; Lin and Murase, 2007), and can be summarized as follows:

1. The decomposition temperature and the char (wt %) of AAC and PACB for the heating rate of 5 ˚C/min were lower than those at 40 ˚C/min. Regardless of the different heating rates, both the decomposition temperature and the char (wt %) of PACB were higher than these of AAC. Compared to the char results of CCA and ACQ, the char of PACB was higher than that of ACQ and lower than that of CCA.
2. The results of the IR response and the TGA–IR spectra from the TGA–IR tests showed that at a heating rate of 5 ˚C/min, the main evolved species, the concentrations of CO_2 and the evolved specie of $=CH_2$ groups, of AAC were less than those of PACB, but at 40 ˚C/min the results were reversed. Compared with the results of CCA and ACQ, the absorbance of the CO_2 peak of AAC and PACB was higher than that of CCA, but lower than that of ACQ.
3. The results of the combustion emissions showed that the highest emission quantity of CO was about 94.0 ppm for AAC, and 120.0 ppm for PACB, and they were both higher than CCA and ACQ. The emission quantity of SO_2 for each sample type, including AAC, PACB, CCA and ACQ, was zero. The maximum NOx for AAC was about 49.5 ppm, and the value was higher than that for ACQ and CCA, but it was insignificant for the amount of PACB during the combustion.

It is evident from the above results that the use of TGA in combination with TGA–IR analysis for analyzing the thermal properties of preservatives has led to useful results, regarding the identification of thermal decomposition products (evolved species and char). The CEM analysis enabled us to determine the emission gas concentrations of O_2 and CO_2, the emission contents (CO,

SO_2, NOx), and the temperature of the emission gases during the combustion of the preservatives.

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