

The Effect of Ammonium Sulphate on Fire Retardancy and Thermal Decomposition Mechanism of Rubberwood by LOI and TGA/DTG

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Abstract

The objective of this research was to investigate the effect of ammonium sulphate (AS) contents having concentration of 0-30 % wt impregnated on rubberwood. For this purpose, thermal degradation mechanism of samples was characterized by thermogravimetric analysis under air whereas the flammability test was applied using limiting oxygen index (LOI) and, reachability was carried out according to JIS K 1571. From TGA/DTG, the impregnation of rubberwood with AS was able to shift the decomposition temperature to the lower side temperature (215 °C) and reduced the degradation rate from 11.48 to 5.45 % °C⁻¹, and the decomposition range of modified wood was wider than unmodified wood. The concentration levels of AS fire-retardant agent affect to change the degradation reaction of wood as a positive effect in term of a high residual weight, a low weight loss, a low initial temperature. The weight residue at 600 °C are found to increase from 0.82 to 3.23 %. Especially, The LOI values of all impregnated samples with AS are classified as self-extinguishing rubberwood, and LOI increased from 26 to 42. This is indicated that the AS impregnation improves the fire retardant property of rubberwood. In addition, the leaching of AS from samples was in range 3-4 % indicated that the good fixation of AS in wood structure. Consequently, rubberwood impregnated with AS can be used as a material where fire retardancy is required.

Keywords: Rubber wood material, leachability, fire retardancy, ammonium sulphate, LOI, TGA/DTG

1. INTRODUCTION

The rubberwood (*Hevea brasiliensis*) is an economically important product of Asia. More than 80 % of total rubber plantation areas are in Asia and accounts for about 30 % of all timber product exports in Malaysia and 60 % in Thailand [1]. Especially, the rubberwood is expected to maintain a high share of wood product exports together with wood from other forest plantations. In addition, it is beautiful and uniform color (white to pale yellow tint), good machining properties, acceptable durability, pleasant appearance and ease in finishing. Furthermore, the physical and mechanical properties of rubberwood are almost comparable with the other commercial timber density 640 kg/m³ and side hardness 4320 such as Dark Red Meranti (*Shorea platyclados*), Sepetir, Nyatoh and Ramin [2]. Although rubberwood is a material with many advantages for residential and non-residential construction, there are still treasures the disadvantages. One of the limitations of wood is the flammable material which be destroyed under the right conditions by heat enough [3]. When heating wood, the wood burning cycle start to hydrolyze, oxidize, dehydrate, and pyrolysis forming a mixture of volatiles (flammable

gases/non-flammable gases), tarry compounds, char and heat releases, and heat must be transferred back to the system until the raw material runs out. Certainly, this situation would cause injuries and accidents. Therefore, the development for fire-retardant wood should drive for safety material. A number of scientists have investigated the reduction of fire performance of wood as ignitability, heat release and the spread of flames with chemical [4, 5].

To reduce the flammability of the wood, chemical agents are loaded into wood structure can lead to modifies the reaction mechanism and pyrolysis product during burning to delay or even extinguish the combustion [6]. Therefore, the investigation on the pyrolysis process of wood treated with fire retardants can help to understand the mechanism of fire retardant efficiency. This above-mentioned chemical is defined as fire retardant. Furthermore, the effective in producing flame retardancy (FR) of treated woods will depend on the type of chemicals, the weight percent gain of chemical, the species of the wood and the impregnated processes. There are researchers previously have been studied various chemical agent to improve the flame retardancy of wood such as inorganic salts, borax, boric acid, monoammonium

phosphate, diammonium phosphate, zinc chloride, ammonium sulphate [7, 8].

Ammonium sulphate (AS) is one of the most effective fire retardants. Mostashari *et al.* [9] investigated the effect of AS treated cotton fabric on the thermal degradation by TGA. They found that the incorporation of AS influenced the decrease of flammable volatiles by shifting first decomposition temperature and more char formation during a heating process which occurred in accordance with gas dilution theory and chemical action theory. The char may act as a thermal barrier, reducing heat transfer to the interior of the material and, thereby, inhibiting the rate of decomposition. It was reported in the literature that the effect of AS on flame retardant of rayon fiber from bamboo wood [10], fir wood [11], sugar-cane bagasse [12], cellulose [13], *Pinus halepensis* needles and their main components (cellulose, lignin and extractives) [14]. Maoshen Chen *et al.* [15] studied the thermal behavior of reconstituted tobacco sheet treated with ammonium salts. Moreover, Statheropoulos and Kyriakou [16] presented that AS release sulfuric acid and NH₃ upon heating at about 284 °C, and sulfuric acid act as a catalyst for the dehydration reaction and promotion of charring and NH₃ act that inhibits gas phase combustion. José Pérez *et al.* [17] has concluded that AS is widely used as a fertilizer or as a fertilizer component. This is the principal components of the powder material utilized in multipurpose of fire extinguishers [18]. The toxicity and eco-toxicity safety data for AS show the chemical agent to be harmless with an acute toxicity: LD₅₀ > 2800 mg/kg (rat) [19]. However, the effects of AS impregnated rubberwood on the thermal degradation during pyrolysis have not yet been extensively investigated to use in wood construction. Therefore, the main compound in the FR agent used in this study is AS. Finally, one limitation of use for AS impregnated wood is unstable in contact with water or humidity during using terms [20]. For these reasons, it is necessary to report results of leachability concerning the effects of such treatments on AS fixation in rubberwood in this study.

For evaluating the effectiveness of wood and wood based materials on the fire performance and thermal properties, there are various method with bench-scale test and large-scale test for research purposes and fire safety classifications for end-utilizations [21]; such as TGA [22], cone calorimetric [23], fire tube test, lateral ignition, tunnel flame-spread tests, LOI tests [24], smoke production tests, flame spread test (LIFT), the single burning item test. Especially, TGA provides a good method for screening the fire retarding effectiveness of several chemicals [25]. LOI measurement has been developed to determine of the relative flammability of wood and the low oxygen index level means that highly flammable materials [26]. A number of scientists have confirmed a suitable and precise indicator of the relative flammability of wood by using LOI value [6, 8, 24, 27].

Both TGA and LOI require a small size that is a simple, convenient and fast method for research purposes [24].

In this research is directed towards investigating the effect of ammonium sulphate with various concentrations of impregnated rubberwood samples on the thermal degradation mechanisms from thermogravimetric analysis and differential thermal analysis techniques. The next stage of investigation uses a LOI value to determine the flammability properties of rubberwood impregnated with AS as well as the leachability of AS was studied.

2. MATERIALS AND METHODS

2.1 Materials

The rubberwood lumbers were obtained for this experiment from commercially produced flat sawn lumber supplied by a local sawmill in Thailand. Samples were carefully chosen for having the same annual ring. The rubberwood timbers were kiln dried at 60 °C and thereafter conditioned to stable moisture content (MC) and then it was machined into samples with the dimensions of 100 (longitudinal) x 10 (tangential) x 7 (radial) mm which were selected by the criteria of similar weight (± 0.0001 g). The rubberwood specimens in this study average density 0.7428 kg/m³. Before impregnation, the wood samples were dried in oven at temperature of 105 °C for 24 h, and then keep into desiccator for two weeks and weighted. Ammonium sulphate (NH₄)₂SO₄ ($\geq 98\%$ purity) was purchased from Ajax Finerchem. Various concentrations (%wt) of AS powder (1, 5, 10, 20, 25 and 30 %) were prepared using diluted water before use.

2.2 Impregnation of rubberwood samples

Wood samples were immersed by vacuum-pressure method according to the principals of ASTM D 1413-07 (2007) with the aqueous solutions at 600 mm/Hg for 60 min by vacuum pump [28, 29]. Afterwards, wood samples were removed from the chamber and shake off lightly to make sure the excess solution left on the surface of woods. After the treatment, all the wood specimens were dried until unchangeable weight at 60 °C. Weight percentage gain (WPG) of each condition shown in Table 1 was measured according to Equation (1) as previously mentioned in other studies [30]:

$$WPG(\%) = (W_2 - W_1) / W_1 \times 100 \quad (1)$$

where W₁ is oven-dried weight of wood sample before impregnation (g), W₂ is oven-dried weight of wood sample after impregnation (g).

2.3 Leaching of treated wood specimens

Procedure Leaching test was conducted according to Japanese Industrial Standard (JIS) K 1571 (JIS, 2004) and Chinese National Standard 6717 (2000) [31, 32]. The procedure involves immersing the five specimens per run in a 1,000 ml beaker containing 400 ml distilled water

added in a ratio of 10 volumes of water to 1 volume of wood, stirring with a shaker follower (400–450 rpm) at 27 °C for 8 h, followed by drying for 16 h and then the specimens were stored in desiccator. The percentages of leaching the AS impregnated samples (%L) were calculated from the oven-dried weight of impregnated wood before and after leaching according to the following equation:

$$\%L = (W_2 - W_3) / W_2 \times 100 \quad (2)$$

where W_2 is oven-dried weight of wood sample after impregnation (before leaching) (g) and W_3 is the oven-dried weight of impregnated wood after leaching (g).

2.4 Limited oxygen index (LOI) measurements

The LOI value was defined fire performance of wood samples as the minimum percentage of oxygen for retaining flaming combustion of each sample under test conditions. LOI measurements of samples were carried out by using a Stanton Red Croft instrument, which is vertical burning test. It was applied for evaluation of LOI in accordance with ASTM D2863-10 [33]. The sample is placed with the holder in a gas mixture of oxygen/nitrogen and adjusting the amount of oxygen until flaming combustion of the sample for 2 inches or 3 minutes after the removal ignition source. The five replicate measurements were observed for each value, with relative standard deviation (RSD) lower than 1 %.

2.5 Thermogravimetric analysis

Thermogravimetric analysis (TGA) was carried out on a thermogravimetric analyzer (TGA-50, Shimadzu). The temperature ranged from room temperature to 600 °C at a heating rate of 10 °C/min with a flow of air atmosphere at 20 mL/min. The sample weight was tested about 10 mg [34-36].

TGA is a common technique in which is monitored continuously the weight change of studied samples with temperature change [26]. The components of the sample decomposed with increasing temperature. TGA is used in determining thermal performance, degradation temperature, moisture content of materials, the levels of compounds in material and amount of residue [26, 36]. This leads to characterize materials containing additives at different levels and evaluate the effectiveness of additives (flame retardant). TGA could be observe the effect of degradation during the process in both inert and oxidative conditions [12, 34-38]. In this study, the air was chosen for purge gas in run for two reasons. The first reason is that to study the decomposition behavior of our materials and to evaluate the effectiveness of flame retardant in

improving the fire performance in the functional environment [37]. Secondly, due to the degradation of fire retardants containing material in oxidative shows more difference than in inert condition of weight loss during a heat change as previous research [34, 37].

3. RESULTS

3.1 Weight percentage gain (WPG)

Weight percentage gains are used widely for calculation to evaluate the amount of chemical substance impregnated in the wood structure after treatment [39]. This value has to be considered fixed solid in the wood matrix with monitor before and after weight of wood in treatment process [30]. The WPG were consorted with what claimed for modified wood with chemical. The WPG for each concentration of wood impregnation at ambient temperature are reported in Table 1. The WPG in wood samples increase linearly with the increment of AS concentration. The highest increment of WPG depending on the concentrations was observed on AS treated wood samples as follows the WPG was founded 9.62 at 30 % concentrations. This indicates that the impregnated AS was fixed in the wood matrix. The amount of chemical interrupts thermal degradation of material, thus this increase is a measure of fire retardant [40]. However, At the AS concentration of 1 %, the retained WPG is -0.21 that may be due to diffuse some water-soluble wood extractives and degraded wood components during impregnation process. Therefore, in this study was exposed 5-30 % AS concentration to focus the influence of the AS agents into the wood.

3.2 Leachability

Table 1 shows the percentages of leaching the AS impregnated rubberwood samples (%L) along with standard deviations. %L of all treated samples after leaching test was found in the range 3-4 %, which means there is the mass loss inside wood sample during test due to the AS flame retardants are water-soluble salt [41]. Amount of leachant increased slightly in treated samples as the loading of AS fire-retardant inside wood increased, likely as a result of better AS fixation when the wood was treated with higher concentrations of fire-retardants. A possible explanation might be that the mass loss of treated wood could be related to removal of excess AS fire-retardant or temporary fixed AS fire-retardant. However, it can be observed from Table 1 that the among of AS almost were able to be fixed into wood structure which

confirmed that AS was an effective flame retardant for wood samples under humidity condition.

Table 1 Weight percentage gain, leachability and LOI of non-impregnated and impregnated wood samples with AS

Code samples	Concentration of $(\text{NH}_4)_2\text{SO}_4$ (%wt)	Weight percent gain ^a (%)	L ^a (%)	LOI (%)
RB0	0	-	-	22.8
RBAS1	1	-0.21 (0.35)	-	-
RBAS5	5	2.15 (0.56)	2.88 (0.50)	27.8
RBAS10	10	5.39 (0.70)	3.08 (0.22)	30.0
RBAS20	20	8.12 (1.79)	3.60 (0.96)	33.0
RBAS25	25	8.37 (1.33)	3.51 (0.75)	37.0
RBAS30	30	9.62 (0.66)	4.37 (1.26)	41.7

a: Each value is an average of 10 specimens and the values in the parentheses are standard deviations.

3.3 Thermal decomposition mechanism

Thermo-oxidative decomposition of tested wood was characterized employing thermal analysis. The results of thermogravimetric analysis for rubberwood samples before and after impregnated with AS 10-30 %wt were carried out in air atmosphere from ambient temperature to 600 °C, are presented by TGA curves and their first derivatives (DTG) in Fig. 1 and 2, respectively, and are summarized in Table 2.

Thermal analysis curves of the untreated rubberwood (Fig. 1 and 2) shows three stages of thermal degradation. It was in agreement with the results reported in the literature for degradation of wood [37, 42]. At below 100 °C, the mass loss of the untreated rubberwood in the evaporating state is resulted from evaporation of moisture. The thermal decomposition of rubberwood mainly occurs in the range 215-512 °C with two distinguishable weight loss zones and the final residue is 0.82%, which is due to the ash content [35]. The main decomposition starts at around 215 °C with a low conversion rate. Then the decomposition rate increases rapidly from around 250 °C to the devolatilization peaks (11.8 % °C⁻¹) at 323 °C from which the rate decreases quickly, down to 1.9 % °C⁻¹ at around 360 °C. This marks the end of the devolatilization and the beginning of the char combustion, which has much lower rates than the devolatilization. This stage is related the thermal decomposition as the combustion and char transformation overlap in the ranges where the volatiles are released. The known decomposition temperature range is approximately 200-375 °C for hemicellulose and

cellulose, which are the two major wood compounds [43]. Furthermore, there is a clear shoulder in the devolatilization stage of the untreated rubberwood. This can be due to the fact that hardwood (rubberwood) have the high hemicelluloses content that usually contains more xylan than softwood. It is the most reactive compound in this temperature range [44]. In the second peak (the char combustion stage) may be attributed to the degradation of lignin and occurs continuously char oxidation [45]. The peak at 450 °C between 375 and 500 °C and the char combustion rate peak (-3.8 % °C⁻¹) are due to oxidization of carbonaceous residues within inorganic solid particles (without volatiles) [44]. The temperature range was in agreement with previous literature [43].

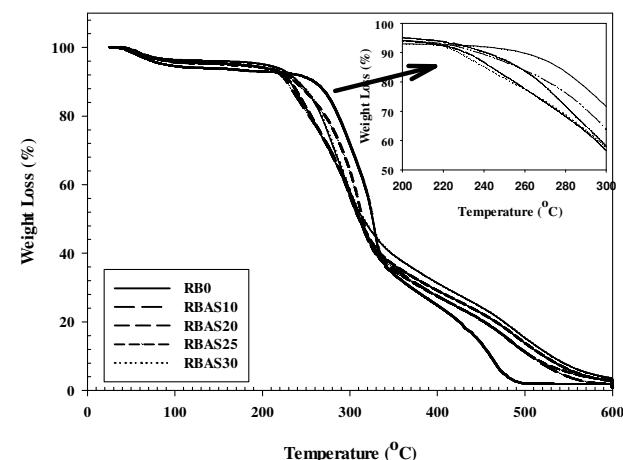


Figure 1 TG curves of the non-impregnated and impregnated rubberwood samples with AS in air atmosphere

Comparison of TGA results for rubberwood and flame-retarded rubberwood (10-30 %AS) presented in Fig. 1 and 2. There were significant difference trend for thermal decomposition behavior of wood impregnated with AS, comparing to the untreated wood. It became clear that AS disturbs the combustion of rubberwood. The mass loss phenomenon was divided into three stages (evaporating stage, devolatilization stage and char combustion stage). The first stage is evaporating stage. One of the most obvious differences between untreated and AS-treated wood in the devolatilization stage (second stage) was the decrease of initial decomposition temperature. It was observed that AS-treated wood started a rapid decomposition after higher concentration treatment that is a lower initial temperature than the untreated wood (225 °C) about 25 %. This is due to the action of AS catalyzes the decomposition of minor consist of wood (volatile and hemicellulose) and produces partial volatile gases

(according to reactions (3) and (4) , before ignition temperature is reached [16, 24]. The volatile gases is mainly NH₃ (non-combustible gases), causing to dilute oxygen concentration in thermal degradation oxidation process [16]. For the reasons above, AS is significant in producing effective flame retardant properties.

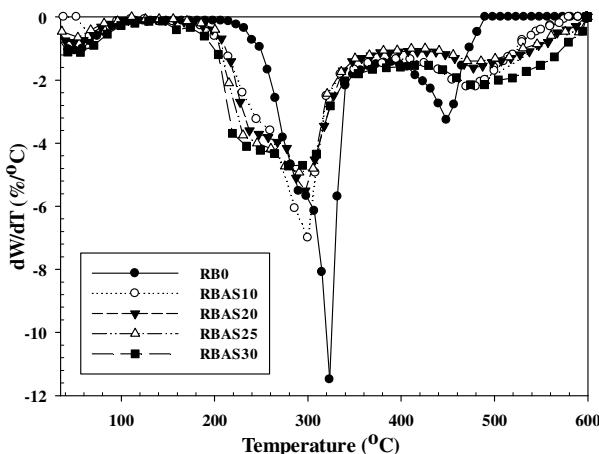


Figure 2 DTG curves of the non-impregnated and impregnated rubberwood samples with AS in air atmosphere

Additionally, there is a shoulder curve in the devolatilization stage of the treated rubberwood wider than the untreated rubberwood and the decomposition rate of AS-treated wood decreased almost three times (4.2 % °C⁻¹) compare with the untreated wood ones (11.8 % °C⁻¹). This indicates that the maximum reaction rate of untreated wood occurs at lower temperatures than for AS-treated wood. A possible explanation could be that the more residue released after decomposition acted both as a thermal insulation barrier and as a barrier for oxygen and flammable volatile products with a higher temperature [9, 12]. The last point in this stage, the temperature of maximum mass loss rate (T_{peak}) of the 30 %AS-treated wood is 234 °C, which is less than 323 °C of the untreated wood. The temperature of maximum mass loss rate

decreases from 300 to 234 °C with the increase of AS concentration (10-30 % AS). This indicated that AS mainly attributes disturbance of the rubberwood combustion due to the above reasons.

At above 400 °C is mainly the third stage, which is supposed to be the decomposition of the char (the char combustion stage). The AS-treated wood results in decreased char combustion rate (47% decreased) and in increased the combustion temperature range compared to results of control samples. The decomposition process is slower with the increasing of AS. This result could be related to the decomposition mechanism is getting more complex when retardants are applied on rubberwood according to reactions (5) [9, 12]. Finally, the measured weight residue at 600 °C for the untreated rubberwood was 0.82 %, whereas for the 10 % AS-treated wood was 2.25 %. Furthermore, when the mass concentration increased from 10 to 30 %, the weight residue value increases from 2.25 to 3.23 % that 42.56 % were increased. This is indicated that concentration level of fire retardant agent affect to change the char combustion mechanism, contributing to the increasing of the amount of the residue [22]. According to the above analysis, it was noted that the incorporation of ammonium salt into wood could retard the thermal decomposition and combustion of the major components of wood [12, 22].

The effect of AS on fire retardancy can be explained possibly by their thermal decomposition with two steps by Statheropoulos et al. [16] . It has been shown that ammonium sulphate decomposes in the first step about at 284.3 °C to produce NH₃ and sulphate acid according to the reactions (3) and (4) . The second step proceeds through the reaction (5) at 383 °C leading to the formation of SO₃ and water in gases phase.

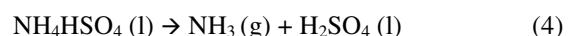
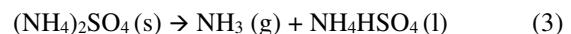


Table 2 Analytical data of the non-impregnated and impregnated rubberwood samples

Sample code	Evaporating			Devolatilization			Combustion			Residue (% w/w)
	T _{min} (°C)	Weight Loss (%)	T _{peak} (°C)	T _{min} (°C)	Weight Loss (%)	T _{peak} (°C)	T _{min} (°C)	Weight Loss (%)	T _{peak} (°C)	
Control	32	8.8	66	214	66.75	323	390	24.45	450	0.82
RBAS10	35	7.15	66	170	70.15	300	419	22.70	468	2.25
RBAS20	31	7.89	59	168	70.73	297	437	21.38	477	2.30
RBAS25	36	8.26	53	167	68.12	289	441	23.62	478	2.96
RBAS30	30	9.24	56	159	65.64	234	429	25.12	489	3.23

T_{min} is the initial decomposition temperature in each stage.

T_{peak} is the temperature of maximum mass loss rate in each stage.

3.4 Flame Retardant Performance

The fire performance of AS-impregnated wood sample was evaluated base on limiting oxygen index measurements [27, 40]. As shown in Table 1 and Fig. 3, the LOI value of untreated rubberwood specimens was found to be 22.8 % that can be categorized as slow burning in air with the classification of material according to ISO 4589 in Table 3 [46-48]. With the adding of AS in the range of 5, 10, 20, 25 and 30 %wt, the LOI values were increased to 27.8, 30, 33, 37 and 41 %, respectively. These LOI values tended to be raised with increasing WPG of AS in wood sample, and all treated samples showed higher LOI levels than controls. This suggests that the combustibility of the wood treated with chemicals decreases and flame retardancy increases.

Table 3 Classification based on LOI values according to ISO 4589 [47]

LOI (%)	Classification of fire
LOI < 20.95	Flammable
LOI = 20.95	Marginally stable
21 < LOI < 28	Slow burning
28 < LOI < 100	Self-extinguishing
100 < LOI	Intrinsically non-flammable

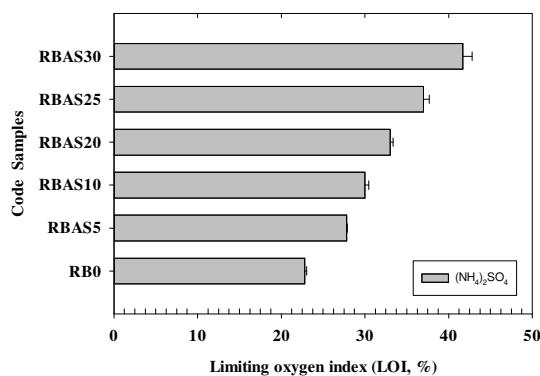


Figure 3 LOI of the impregnated rubberwood samples

It was indicated that the higher the LOI value can increase the flame retardancy of the impregnated woods [8]. The weight percent gain of fire-retardant wood can reach to 9.5 %, while the LOI value could be up to 42 %. Especially, all treated rubberwood samples are classified as self-extinguishing that mean the wood samples will stop combustion after the removal ignition source. This is appropriate to play a role to achieve this goal. As a result, AS treated rubberwood can be considered as suitable flame retardants for wood-based materials, such as interior structures and furniture for buildings. Experiments conducted by TGA/DTG and LOI measurement present that ammonium sulphate impregnated into rubberwood significantly improved the fire performance.

4. CONCLUSION

The present work presents the impregnation of flame retardant ammonium sulphate improved significantly the properties of rubberwood targeting high quality utilization. With vacuum-pressure method, the WPG in wood increase linearly with the increment of AS concentration. The WPG could reach to 9.5 %, while the LOI value could be up to 41.7 %, and LOI still increased from 27.8% to 41.7% than untreated wood (22.8%). This value can be classified to be a self-extinguishable wood, which indicated AS can improve the fire retardancy of rubberwood for potential as a construction material. The leachability test indicated that AS still be fixed into wood structure after test which confirmed that AS can be used under humidity condition. TGA in an oxidation atmosphere results showed that the addition of AS changed the initial decomposition temperature (about 25 %), shifted the main decomposition temperature from 323 to 279 °C, increased the temperature range of decomposition, accelerated main decomposition and improved the residue.

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7. BIOGRAPHIES



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