

# Studies on Metallurgical Coke Production from Woody Biomass

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## 論文内容の要旨 Thesis Summary

Due to growing global environmental problems, which is often represented by the global warming, moving away from fossil fuels in favor of reliance on renewable sources is an inevitable trend to develop continuously and intensively. Among renewable resources, woody biomass is an abundant resource on the earth that has been used as an alternative replacement for portions of the fossil fuels for production of power/heat, iron, chemicals and materials. Many researchers have therefore devoted extensive efforts to developing technologies of woody biomass upgrading and conversion in these decades. Selective and efficient transformation and utilization of woody biomass are restricted by its inherent properties. For example, many researchers tried to produce coke through addition of woody biomass to coal, but none of them could produce high-strength coke. Rather, they found negative impacts of adding woody biomass on the strength of resulting coke. Only one successful example is high-strength coke production from hydrothermally-treated woody biomass, while this type of treatment is not economically feasible.

The author has developed novel biomass pretreatment methods that successfully yield high-strength blast furnace coke from biomass alone and also biomass-coal blend at any mixing ratio. This thesis focuses on (1) the development of sequences of torrefaction of woody biomass, pulverization, binderless hot briquette and carbonization, (2) that of a method to prepare formed coke from mixture of torrefied biomass and non-caking coal based on (1) at any mixing ratio (i.e., 0–100 mass% of torrefied biomass in the mixture), (3) that of modification (control) of coke reactivity by leaching alkali and alkaline earth metallic (AAEM) species out of the torrefied with acetic-acid-rich water that is produced simultaneously with the torrefied biomass.

**Chapter 1** of this thesis introduces the background, current status of woody biomass utilization including types of biomass, biomass conversion technologies and some details and technical problems of a particular technology, *i.e.*, torrefaction.

**Chapter 2** describes the results of production of high strength coke from torrefied biomass by employing a self-developed novel laboratory scale experimental set up. Chipped and pre-dried Japanese cedar was heat-treated in inert atmosphere at 225–325°C ( $T$ ), pulverized to sizes in three different ranges, molded into briquettes (in form of thick disk with diameter/thickness  $\approx 2.5$ ) at temperature up to 200°C by applying mechanical pressure of 128 MPa. The torrefied/briquetted cedar

(TBC) was then converted into coke by heating to 1000°C in inert atmosphere at normal pressure. This process sequence enabled to prepare coke having indirect tensile strength ( $S_t$ ) of 8–32 MPa and apparent density of 0.90–1.13 g/cm<sup>3</sup>. The torrefaction greatly improved pulverizability of the cedar, which was further promoted by increasing  $T_t$ .  $S_t$  of TBC and that of coke both increased as the particle sizes of TBC decreased, but this explained only a very minor part of significant effect of  $T_t$  on  $S_t$  of the coke.  $S_t$  was maximized at  $T_t = 275^\circ\text{C}$  regardless of the degree of pulverization. The  $T_t$  effects on physicochemical properties of TBC and coke were investigated in detail. The difference in  $S_t$  of coke by  $T_t$  was mainly due to that in the increment of  $S_t$  along the carbonization at 500–1000°C. Fracture surfaces of the coke had particular morphologies that had been inherited from the original honeycomb structure of the cedar.

**Chapter 3** describes result of the coke production from mixture of torrefied cedar (TC) and non-caking coal by a sequence (I) of pulverization to sizes < 100  $\mu\text{m}$ , mixing, binderless hot briquetting and carbonization, or another sequence (II) that replaced the pulverization and mixing by co-pulverization to sizes < 40  $\mu\text{m}$ . Sequence I produced coke with tensile strength of 5–17 MPa from TC-coal mixtures over the entire ranges of TC mass fraction in briquette of 0–100%, torrefaction temperature ( $T_t$ ) of 250–300° C, and choice of coal (sub-bituminous or medium-volatile bituminous coal). The mixing of TC and coal hindered densification of coke due to hindrance of shrinkage of more-shrinkable TC-derived particles during the carbonization under many of the conditions. Nevertheless, positive synergy occurred in the coke strength at TC mass fractions of around 75%, where coal-derived particles were dispersed in the matrix of TC-derived particles, bonded to them during the carbonization, and behaved as a reinforcement of the matrix. The bonding between TC-derived and coal-derived primary particles was revealed by scanning electron microscopy. Sequence II gave cokes having strengths as high as 22–28 MPa. The fine pulverization increased frequencies of mutual bonding of TC-derived particles, that of coal-derived particles, and bonding between TC-derived and coal-derived particles per coke volume. The strength of coke from TC-coal mixture generally followed volume-based additivity of strengths of cokes from TC and coal. This was realized by mixing primary particles TC and coal within a  $\approx 10 \mu\text{m}$  scale or even smaller.

**Chapter 4** reports the experimental results of the leaching of AAEM species with simulated water phases (rich in acetic acid) of bio-oil from the torrefaction, demonstrating increase in the tensile strength of coke from the torrefied cedar from 16.6 to 21.3 MPa and decrease in its reactivity with CO<sub>2</sub> by factors of 5–40. In this chapter are discussed the mechanism that caused such changes in the coke properties. The lowered reactivity was due straightforwardly to significant loss of *catalytic* AAEM species (mainly, K, Ca and Mg) and by the leaching. The increased strength was explained by slightly enhanced softening (thermal plasticization) of the torrefied biomass in the early stage of carbonization and enhanced shrinkage in the middle to late stage.

**Chapter 5** summarizes the findings, described in detail in the preceding chapters.