NO.1

Year 2023	**Summary of Thesis**

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(Title)
Basic research for low-energy production of chitin/chitosan boards

Japan relies on imports for resources because it does not produce much in the way of petroleum and ores. Domestically, however, Japan has abundant forest and ocean resources. Chitin and chitosan are of particular interest. These are natural polysaccharides obtained mainly from marine products such as crab and shrimp shells, and are the second largest producer after cellulose. In a previous study, cellulose and starch were hot-pressed to create a citric acid cross-linked foam material. Since cellulose and starch and chitin and chitosan are both based on pyranose, so this experiment is expected to show a similar response, but what was created was board material. In the previous experiment, the temperature of the hot press was set at 220°C, but here, the temperature was set at 90°C, and plates were created. We received a lot of interest in this low-temperature hot-pressing method at academic conferences, etc., and we thought that it could contribute to the low energy consumption of material synthesis, so we explored the possibility of developing chitin/chitosan plates at low temperatures in this study.

In this study, a paste was prepared by mixing 1 g chitin, 3 g chitosan, 0.2 g citric acid, and 0.1 g sodium hypophosphite hydrate, and adding 20 ml pure water. This was poured into a JIS 7139 mold and hot-pressed at 90°C and 40 MPa. This sample is designated as LT (Low Temperature). As a control experiment, a sample of the same paste as LT was pressed without heating (RT : Room Temperature), a sample of LT without citric acid and sodium hypophosphite-hydrate, which are cross-linking agents, was pressed without heating (PO : Pressure Only), and a sample of LT with the same paste as LT, but without heating (RT : Room Temperature) was pressed without heating. A hot-pressed sample (NCA: Non Cross-linking Agent) without the cross-linking agent was prepared for the purpose of FT-IR measurement, and evaluated

Graduate School of Science and Technology, Chitose Institute of Science and Technology

NO.2

by FT-IR, 13CNMR of CP/MASS, three-point bending test, tensile test, SEM, and so on. In addition, samples were prepared by controlling conditions such as synthesis temperature and materials used as appropriate for each analysis method.

As for the results, first, a new peak around 1716 cm-1 and 1735 cm-1 was found in the ATR analysis of FT-IR in the experiment at 220°C, the condition tested in the previous study, but no distinct new peak could be found in any of the samples prepared at 90° C. In addition, solid-state NMR did not reveal any spectral differences between the addition of crosslinking agents and heating.

Therefore, a three-point bending test was performed on three different samples, PO, RT, and LT, and statistical analysis showed that there was a significant difference between LT and the other samples as shown in Fig 1. A hint for a cross-linking interaction is that the LT sample cannot be dispersed in water.

In consideration, the significant increase in stress with the addition of the cross-linking agent and hot pressing is thought to be due to the new formation of some bond other than the ester bond, which at this time is thought to be an amide bond. However, since FT-IR and NMR did not reveal any new peaks, a new analytical method is desired. The reason for the significant increase in stress and dispersion in water at RT may be ionic bonding between citric acid and ammonium groups, but a new analytical method is also desired to elucidate the mechanism of strength increase.

In general, it was found that stress strengthening is possible even at low temperatures by experimental methods, but the chemical mechanism and mechanism of this phenomenon remain to be elucidated, and future research is expected.

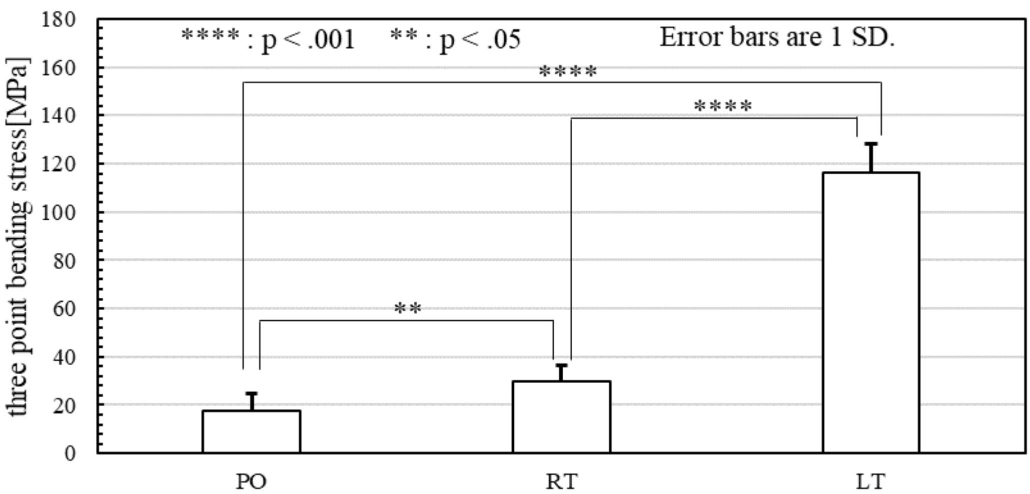

Fig 1. Multiple Comparison Result of Bending Stress

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