

UNIVERSITI TEKNOLOGI MARA

**THE EFFECTS OF EDTA ON THE
ISOLATION OF CELLULOSE FROM
BANANA STEM USING
ATMOSPHERIC SODA PULPING**

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ABSTRACT

The pulp and paper industry, which produces various types of papers, packaging materials, and household products, uses over 40% industrial wood traded globally. Non-wood cellulose isolated from agricultural residues, such as banana stems, offers an alternative to the limited supply of wood and environmental impacts of deforestation. Soda pulping using sodium hydroxide (NaOH) is the most basic chemical treatment that efficiently removes lignin; however, simultaneous hydrolysis of cellulose cannot be avoided. According to previous studies, ethylenediamine tetraacetic acid (EDTA) works as an excellent additive to improve the pulp yield, but its influence on lignin removal and cellulose properties is hardly mentioned. Therefore, the efficiency of NaOH/EDTA for lignin removal, pulp yield, and cellulose content was evaluated in this study. The pulping process was optimized using response surface methodology (RSM) and the effects of the anticipated esterification between EDTA and cellulose on the cellulose structure were analyzed. NaOH/EDTA pulping was performed at atmospheric boiling temperature of 100 ± 5 °C due to the low lignin content of $15.3\pm 0.1\%$ in the banana stem. The liquid-to-solid ratio (L/S) was fixed at 10 ml/g for complete immersion of the sample. An analysis of variables based on one-factor-at-a-time (OFAT) design was carried out to determine the suitable range for optimization. The pulping duration was varied from 30 to 180 minutes, NaOH charge was varied from 14% to 20% w/v, and EDTA charge was varied from 0% to 10% w/w. A five-level central composite design was adopted during the optimization of the NaOH and EDTA charges for maximum lignin removal, pulp yield, and cellulose content. Esterification was evaluated using Fourier transform infrared (FTIR) analysis and the degree of substitution (DS). Cellulose hydrolysis was monitored by measuring its degree of polymerization (DP). Surface morphology, crystallinity, and thermal stability were observed using scanning electron microscope (SEM), X-ray diffractometer (XRD), and thermogravimetric analyzer (TGA), respectively. The finding suggested that an efficient lignin removal of 61.5% and cellulose content of 89.9% was achieved at a short pulping duration of 30 minutes with a high NaOH charge of 20% w/v and EDTA charge of 10% w/w. However, a maximum pulp yield of 66.2% was achieved using a low NaOH charge of 14% w/v and high EDTA charge of 10% w/w. At optimized 17.7% w/v NaOH and 10% w/w EDTA, the addition of EDTA has improved the lignin removal by $7.0\pm 2.4\%$, pulp yield by $18.5\pm 2.1\%$, and cellulose content by $0.6\pm 0.3\%$ as compared to a control sample treated without EDTA. A small substitution of 0.11 ± 0.01 occurred due to the esterification between EDTA and cellulose, as determined from the DS and FTIR analyses. The potential of EDTA in suppressing the hydrolysis of cellulose was supported by a higher DP of 2140 ± 55.3 for the NaOH/EDTA-treated sample as compared to the NaOH-treated sample (1907 ± 62.9). FTIR analysis also validated the successful removal of lignin, which was further supported by a smoother fiber surface and separation of cellulose microfibrils, as observed from the SEM micrographs. Furthermore, esterification of cellulose with EDTA disturbed the hydrogen bond network in cellulose and consequently lowered its crystallinity index and degradation temperature. Based on these findings, it was concluded that the addition of EDTA at the optimum conditions for this study successfully improved the pulp yield at minimal cellulose hydrolysis, but the substitution should be controlled as to not severely affect the properties of cellulose.

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CHAPTER ONE

INTRODUCTION

1.1 Research Background

Wood, either hardwood or softwood, is the main source of cellulose in the pulp and paper industry. According to the data from Hansen *et al.* [1] accessed through the Global Forest Watch [2], an online forest tracking and alert system, approximately 437 million hectares of tree cover loss were recorded globally from 2001 to 2021 with 8.67 million hectares recorded in Malaysia alone. Figure 1.1 shows the tree cover loss around the world and specific data with regard to Malaysia. Although deforestation activity has reduced since 2016, approximately 278 thousand hectares of Malaysian forest were cleaned in 2021, which is equivalent to 135 million tonnes of carbon dioxide (CO₂) emissions. The limited supply of wood and growing awareness of the impact of deforestation have contributed to commercialization of non-wood cellulose.

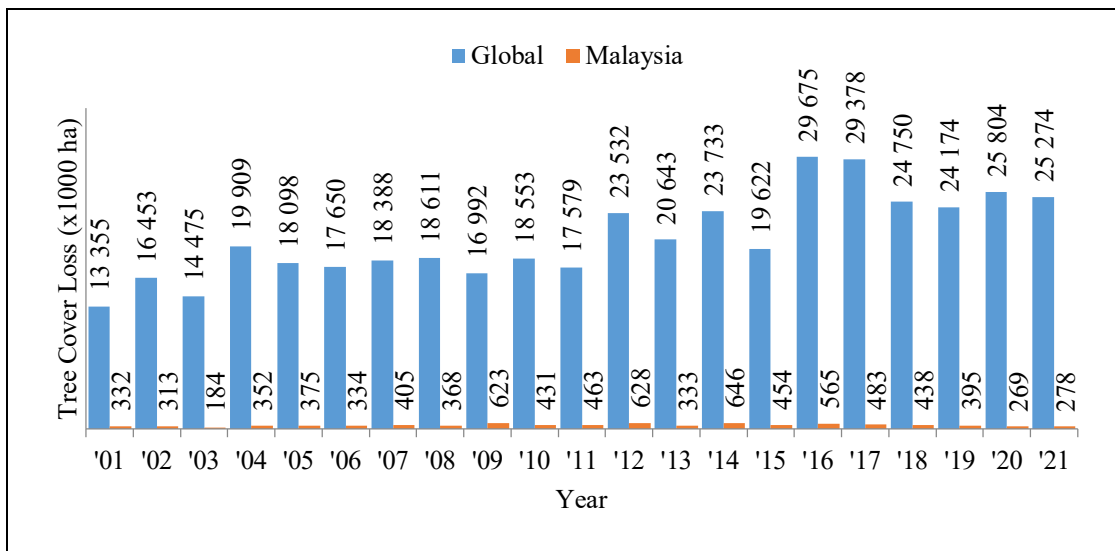


Figure 1.1 The Global and Malaysia Tree Cover Loss From 2001 to 2021 [1, 2]

Non-wood sources are often considered an excellent alternative to wood because they contain a similar amount of cellulose, yet lower in lignin, which allows for processing at lower energy and chemical consumption (see Table 2.1). Among the commonly used non-wood sources for the production of pulp and paper are bagasse, straw, bamboo, and old newsprint [3].