Lipase Treatment for Pitch and Stickies Control in Recycled Pulps: Application in a Duplex Board Mill

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This study evaluated whether lipase enzymes could control pitch and stickies contamination in recycled pulps from a duplex board mill. The tacky contents of four recycled pulp types—old magazine paper (OMG), old book paper (OB), old newspaper (ONP), and old corrugated containers (OCC)—were analyzed using Sudan IV staining and image analysis of handsheets. The reaction times of lipase at varying dosages on pitch and stickies were monitored up to one hour under controlled conditions. The tacky contents, quantified in terms of total stained area and particle count, were substantially higher in OB and OCC than in ONP and OMG, with the latter showing the lowest contamination level. Lipase activity was most effective within the first 30 min. Thereafter, the area reduction of tacky materials plateaued, and the larger aggregates fragmented, slightly increasing the particle count. It was inferred that a 30-min treatment time optimally balances efficiency and cost. Lipase treatment effectively reduced the pitch contents in both OMG and OCC pulps but was less efficient in OMG. The enzyme activity was apparently inhibited by the ink-derived compounds in OMG, but it was enhanced by the many lipase-accessible lipids in OCC, enabling a stronger, dose-dependent response. Lipase emerged as a promising biotechnological solution to pitch control in a duplex board mill.

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Keywords: Pitch deposit; Lipase; Recycled pulps; Duplex board mill; Sudan IV; Staining; Image analysis

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INTRODUCTION

The paper industry relies on pulps sourced from both wood (virgin pulp) and post-consumer waste papers (recycled pulps) (Li *et al*. 2023). Although recycled pulps enhance the environmental sustainability and resource efficiency of papermaking, they introduce contaminants and process residues that are difficult to remove (Suciu *et al*. 2013; Chang *et al*. 2019). Among the most persistent and detrimental problems in both virgin and recycled pulp processing is the formation and deposition of pitch (Gutiérrez *et al*. 2001; Rivera 2004), an umbrella term for hydrophobic substances primarily composed of triglycerides, free fatty acids, sterols, waxes, and other extractives from wood resins (Qin *et al*. 2003). Recycled pulp presents additional challenges beyond pitch. It contains sticky foreign matter residues originating from additional hydrophobic contaminants such as adhesives, printing inks, and coating agents during the paper recycling process (Tanveer *et al.* 2024).

These substances are collectively referred to as "stickies" (Licursi *et al*. 2016), and their combination with pitch can lead to even more significant papermaking process issues.

Pitch particles and stickies tend to agglomerate and deposit on the machine surfaces, felts, and rollers of papermaking equipment, causing sheet breaks and increasing the frequency of cleaning and maintenance (Lipowski *et al*. 1971; Hubbe *et al*. 2006). Besides reducing the operational efficiency of papermaking, pitch deposits generate spots, holes, and other defects that deteriorate the quality and reduce the commercial value of the final product (Buikema and Naka 1997; Dou *et al*. 2023). Consequently, effective pitch control has become an essential component of modern papermaking operations, which increasingly rely on lower-quality recycled materials (Haynes 2013).

Traditionally, pitch has been controlled through mechanical separation (*e.g.,* filtration, flotation), chemical additives such as fixatives, coagulants, or dispersants, and surfactants that emulsify hydrophobic substances (Lipowski *et al*. 1971). Furthermore, methods employing talc and bentonite have been predominantly utilized (Hayashi 1999). However, these substances present the drawback of potentially remaining in the paper or white water, which can affect the final product or interact with other additives, thereby diminishing the efficiency of the papermaking processes (Lipowski *et al*. 1971; Asselman and Garnier 2000). Although these approaches are temporarily effective, they are often limited by high operational costs, potential environmental concerns, poor long-term effectiveness, and incompatibility with evolving sustainability goals (Skals *et al*. 2008). Moreover, chemical treatments may introduce new contaminants or interfere with the paper properties (Mofokeng *et al*. 2024).

In recent years, biotechnological solutions have emerged as more sustainable alternatives for pitch control (Fischer and Messner 1992). A particularly promising solution is lipase, a hydrolase that catalyzes the breakdown of ester bonds in triglycerides, forming glycerol and free fatty acids (Ramnath *et al*. 2017; Javed *et al*. 2018; Zan *et al*. 2019). This enzyme can selectively degrade pitch components without introducing harmful chemicals (Kontkanen *et al*. 2004). Lipase functions by breaking down the ester bonds in the pitch within the pulp, reducing it to smaller particles and thereby decreasing its stickiness (Ramnath *et al*. 2017). Other potential advantages of lipases are targeted action, low toxicity, and compatibility with various papermaking processes (Liu *et al*. 2012).

Despite their promise, lipase-based pitch control technologies require careful consideration of the enzyme type, source, specificity, stability under the process conditions (*e.g.,* temperature, pH, presence of surfactants), and cost-effectiveness (Sharyo *et al*. 1993; Shu *et al*. 2012). In addition, recycled pulp is a heterogeneous material with variable pitch composition and load, necessitating tailored enzyme formulations and process optimization (Bajpai 1999).

This study investigated the applicability of lipase to recycled pulps obtained from a duplex board mill. Four types of recycled pulp obtained from the Hansol Paper mill were treated with varying amounts of lipase and reaction times to prepare handsheets. Handsheets were then dyed step-by-step using a Sudan IV solution, and the surface images of the dyed handsheets were captured using an optical microscope. The area and number of pitch or stickies particles in the images were analyzed using image analysis software. Next, the effectiveness of lipase as a pitch control agent was evaluated in terms of the total stained area and pitch-particle count on sheets formed from different recycled pulp types.

EXPERIMENTAL

Materials

Recycled pulp furnishes were obtained from Hansol Paper (Daegeon, Republic of Korea), which produces duplex boards. The materials were classified into different types of recycled pulp: old magazine, old book, old corrugated container, and old newspaper. The enzyme (lipase) was supplied by Sunson Industry Group Co., Ltd. (Beijing, China), and its specifications are listed in Table 1. The pitch particles were stained with Sudan IV solution prepared from Sudan IV (powder, Tokyo Chemical Industry, Tokyo, Japan) and ethylene glycol (99.5%, Samchun Chemicals, Seoul, Republic of Korea).

Table 1. Enzyme Specifications

Items	Specifications
Model Name	SUN86
Component	Lipase
Protein count	6.03 mg/mL
Appearance	Liquid with low subsidence
Color	Brown
Odor	Normal fermentation odor
Enzymatic Activity	≥ 2,000 u/mL

Methods

Preparation of Sudan IV staining solution for pitch analysis

Sudan IV can stain neutral fats, fatty acids, cholesterol, and other molecules originating from wood. As a fat-soluble dye, Sudan IV readily dissolves in non-polar lipid molecules, typically producing a red coloration upon staining. Accordingly, Sudan IV solution was employed as the staining agent of pitch components in this study (Nam *et al*. 2015). The extent to which Sudan IV also stains typical stickies materials was not determined. To prepare the staining solution, 0.7 g of Sudan IV powder was dissolved in 100 mL of ethylene glycol with stirring at 100 °C for several minutes. The undissolved dye was subsequently removed with Whatman No. 2 filter paper, producing a clarified Sudan IV solution suitable for staining and image analysis.

Pitch analysis of the recycled pulps from a duplex board mill

Recycled pulp furnishes for the pitch and stickies analysis were collected at the Hansol Paper mill, which produces duplex boards from various waste papers. Four recycled pulp furnishes used in the mill were collected at each machine chest: OMG, OCC, OB, and ONP lines. The original consistencies of OMG, OB, OCC, and ONP (~3% to 4%) were diluted to 0.7% for handsheet preparation in the laboratory. Handsheets with a grammage of 60 g/m²±5 g/m² were then produced following the TAPPI T205 sp-06 procedure (2006). The wet handsheets were pressed at 345 kPa for 5 min under a laboratory wet press (Model 326; Wintree Corporation, Osaka, Japan) and subsequently dried using a cylinder dryer at 120 °C (Daeil Machinery Co. Ltd., Daejeon, Republic of Korea). The prepared sheets were used for pitch analysis.

Effect of enzyme treatment on pitch or stickies content of the recycled pulp sourced from the duplex board mill

The effect of lipase reaction time on control of pitch and stickies was evaluated on OMG. First, the OMG was diluted to 0.7% consistency for handsheet preparation. After adding 0.10% lipase to the oven-dried pulp, the diluted pulp slurry was maintained in a water bath with mixing at pH 7.0 and 35 °C for 1 h. Enzyme-treated pulp was sampled at 10-min intervals and formed into handsheets with a grammage of 60 $g/m^2 \pm 5$ g/m^2 following the TAPPI T205 sp-06 procedure (2006). The wet handsheets were pressed and dried as described in the Pitch Analysis section.

After determining the optimal lipase reaction time, the effect of enzyme dosage on pitch or stickies content was analyzed by adding 0.00%, 0.07%, 0.10%, 0.12%, or 0.15% of the enzyme (per mass of oven-dried fibers) to OMG and OCC furnishes with a consistency of 0.7%. The samples were vigorously mixed in a water bath at pH 7.0 and 35°C for the optimal reaction time, and handsheets with a grammage of 60 $g/m^2 \pm 5$ g/m^2 were then produced following the TAPPI T205 sp-06 procedure (2006). The wet handsheets were pressed and dried as described in the Pitch Analysis section. The pitch contents of the prepared sheets were then analyzed.

Pitch and stickies analysis

The prepared sheets were cut into small pieces and immersed in 50% ethanol. After immersion, the samples were dried with blotting papers and transferred into conical tubes containing the Sudan IV staining solution. Subsequently, they were incubated at 38 °C with agitation at 300 rpm for 24 h to facilitate staining.

Following incubation, the stained sheets were rinsed in 50% ethanol to remove excess dye and then dried thoroughly. The stained pitch in the sheets was imaged under a stereomicroscope at 40× magnification, as shown in Fig. 1. The pitch content was quantitatively analyzed using image analysis software (AXIOVISION, ZEISS, Oberkochen, Germany) and expressed in terms of total stained area and count of pitch particles.

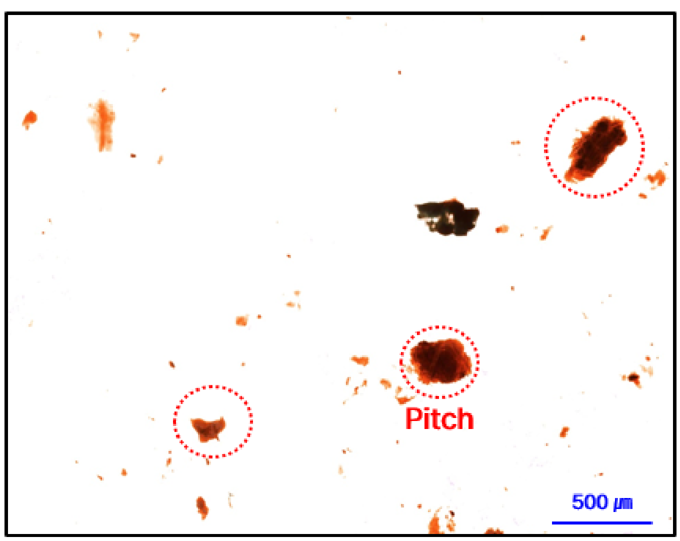

Fig. 1. Microscopic image example of stained pitches in the sheet

RESULTS AND DISCUSSION

Pitch and Stickies Content of the Recycled Pulps from a Duplex Board Mill

Figure 2 presents microscopic images of stained pitch particles in the sheets produced from the four recycled pulps. Pitch components stained with Sudan IV typically appear red. Due to their hydrophobic nature, it is reasonable to expect that synthetic sticky materials in the pulp likewise will take up the Sudan IV dye. Residual pitch and stickies were observed in all sheets, but the amounts depended on the type of recycled pulp. As images alone could not provide a quantitative comparison of the samples, the contents of tacky materials were objectively obtained through image analysis. Figures 3 and 4 show the total area and the number of pitch or stickes particles in the stained sheets made from recycled pulps, respectively. The area was highest in OB, followed by OCC, ONP, and OMG in descending order. The particle count exhibited a similar trend, being highest in OB and lowest in OMG. Interestingly, the particle count was lower in ONP than in OCC.

Pitch in recycled pulp is typically derived from natural resins in softwoods and from various hydrophobic substances such as triglycerides, waxes, fatty alcohols, printing inks, adhesives, and coating agents. The high content of stainable materials in OB and OCC was likely attributable to the elevated presence of sticky contaminants originating from printed and coated paper, adhesives, and other additives commonly found in books and corrugated packaging. OCC and ONP also presumably contain substantial amounts of foreign substances such as ink, pressure-sensitive adhesives, and latex; it appears that these components, when combined with the pitch, increase their tested pitch content. In particular, for OCC, the wax used in coating the raw material, paperboard, is composed of fatty acids, making it possible for it to be stained by Sudan IV. Conversely, OMG is primarily composed of white ledger and magazine-grade papers with relatively few contaminants, which likely accounted for its comparatively low pitch content among the recycled pulps.

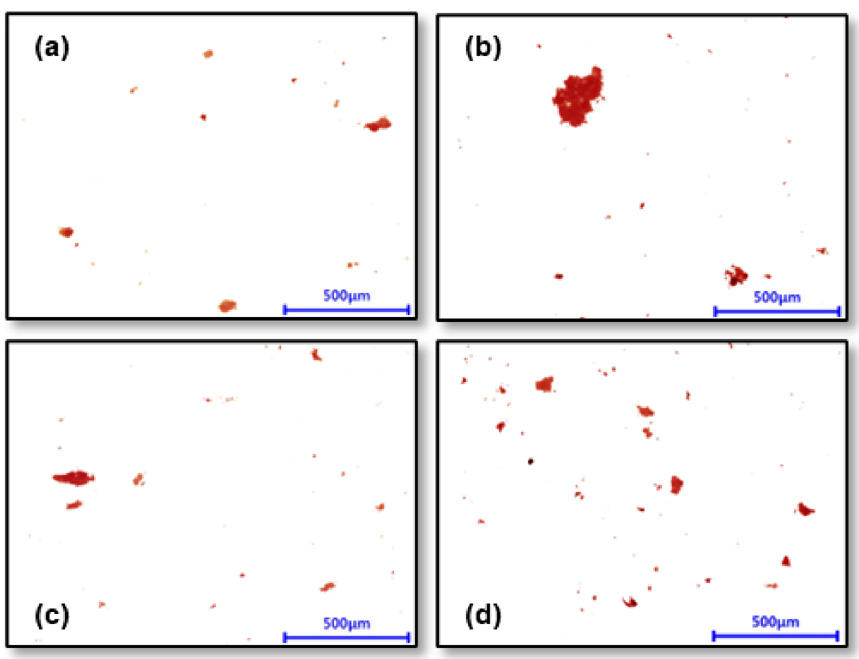

Fig. 2. Microscopic images of stained pitches in the sheets prepared from (a) OMG, (b) OB, (c) OCC, and (d) ONP

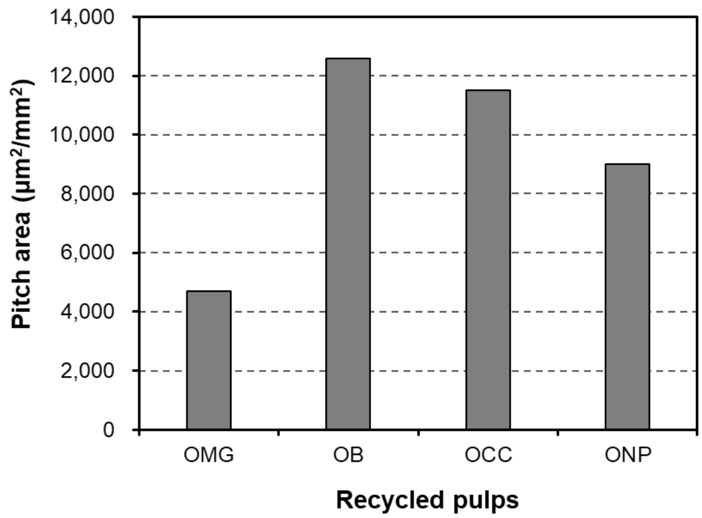

Fig. 3. Pitch area on sheets prepared from the recycled pulps from the duplex board mill

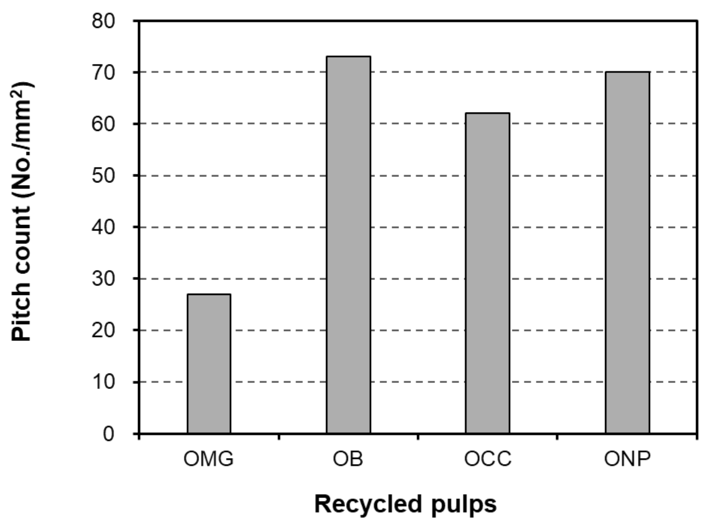

Fig. 4. Pitch-particle counts on sheets prepared from the recycled pulps from the duplex board mill

Effect of the Reaction Time of Lipase on the Measured Pitch Content in OMG

To analyze the effect of lipase reaction time on control of pitch and stickies, the changes in pitch area and pitch-particle count on sheets formed from OMG were determined after 0.10% lipase treatment for various reaction times at pH 7.0 and 35 °C. The results are presented in Figs. 5 and 6.

The area of stained material was highest at the untreated control and gradually decreased with increasing reaction time. The reduction was marked between 0 and 30 min, less prominent beyond 30 min, and stabilized from 40 to 60 min. The plateau suggested that most of the enzymatic activity associated with pitch degradation occurred within the first 30 min; additional treatment time did not obviously reduce the pitch area. The pitch-particle count also substantially declined at 30 min, but it slightly increased between 40 and 60 min. This increase might be attributed to the fragmentation of larger pitch or stickies particles into smaller countable ones, rather than to pitch regeneration.

Therefore, lipase activity was most vigorous within the first 30 min and this reaction time was deemed not only sufficient but also economical for industrial applications.

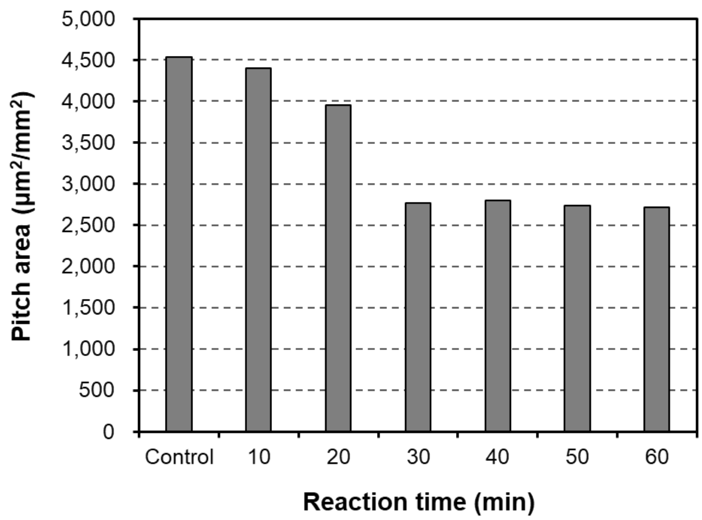

Fig. 5. Areas of stained pitch particles in OMG sheets at different lipase reaction times

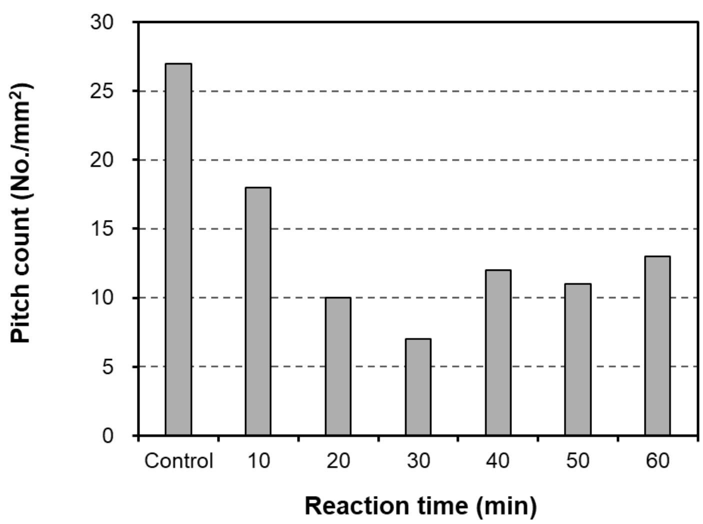

Fig. 6. Counts of stained pitch particles in OMG sheets at different lipase reaction times

Effect of Lipase Dosage on the Pitch Control of OMG and OCC

Figure 7 presents the stained areas on OMG sheets exposed to lipase at different dosages. The pitch area clearly decreased with increasing enzyme dosage, particularly as the dose increased from 0.10% to 0.12%. The reduction of stained area continued at higher doses, but the decline was slight and tended toward stabilization. This trend suggests that the enzyme was most effective when added at 0.12%. Beyond this threshold, the enzyme dosage offered limited improvement.

Figure 8 presents the pitch-particle counts on OMG sheets exposed to various lipase dosages. The 0.10% enzyme treatment remarkably reduced the pitch-particle count but this initial decrease was followed by stabilization at higher enzyme dosages.

Both the stained areas and particle counts confirmed that lipase treatment effectively reduced pitch or stickies contamination in OMG pulp, especially at dosages between 0.10% and 0.12%. Higher enzyme dosages yielded minimal additional benefits.

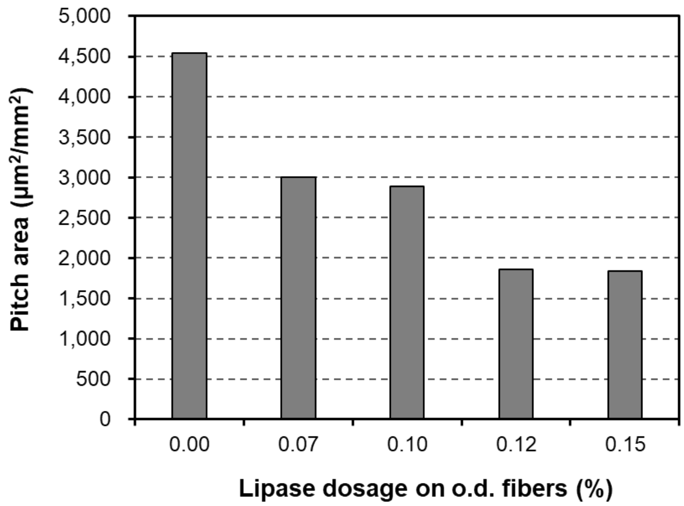

Fig. 7. Areas of stained pitch particles in OMG sheets depending on different lipase dosages

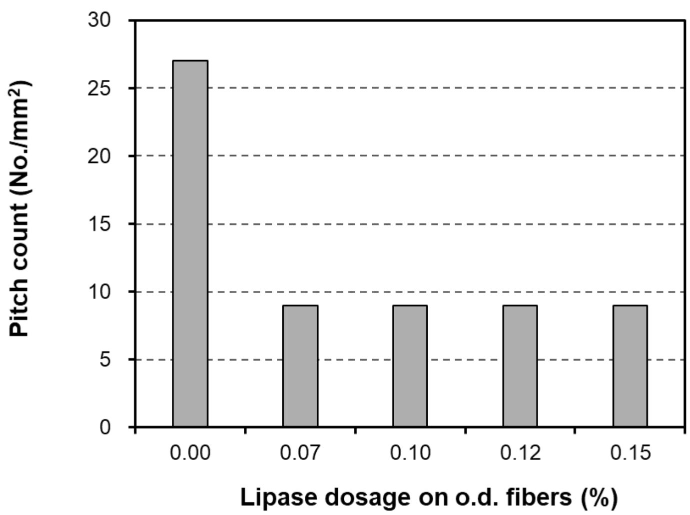

Fig. 8. Counts of stained pitch particles in OMG sheets depending on different lipase dosages

Increasing the lipase dosage also decreased the total stained area on OCC sheets (Fig. 9). The area reduction was notable at a lipase dosage of 0.10% and steadily continued up to 0.15%. This consistent downward trend indicated that lipase effectively hydrolyzed the ester bonds within pitch or stickies substances, thus facilitating the dispersion or removal of pitch and possibly other associated matter from the furnished OCC.

Similarly, the number of stained particles on the OCC sheets decreased with increasing lipase dosage (Fig. 10). The decrease rate was most pronounced between 0% (the untreated sample) and 0.07% and slowed across higher dosages, indicating that most of the easily accessible particles of pitch or stickies were hydrolyzed at lower dosages while the remaining particles were more resistant or structurally embedded.

The deposit control efficiency of lipase at the same dosage differed between OMG and OCC. In OMG, which typically contains printing inks and various additives, the composition of pitch and stickies is complex and chemically resistant. Ink-associated compounds may limit access to the lipid substrates digested by lipase, hindering enzymatic activity. Other compounds comprise non-lipid components that are unsuitable for enzymatic hydrolysis. In contrast, OCC contains a high proportion of resinous wood-derived pitch, such as triglycerides and other neutral lipids, which are susceptible to enzymatic degradation by lipases. In such cases, pretreatment before the application of lipase may be necessary, or a different type of enzyme might need to be employed (Hubbe *et al*. 2018). Additionally, OCC comprises a substantial amount of recycled pulp that has undergone processing involving wax during its manufacturing (Stauffer *et al*. 2001). The wax used in paperboard coating contains fatty acid components and is comprised of ester bonds (Ahuja and Rastogi 2023), which are likely to be broken down by lipase activity. Consequently, lipase treatment effectively reduced the pitch components in OCC, exhibiting a dose-dependent decrease in pitch content.

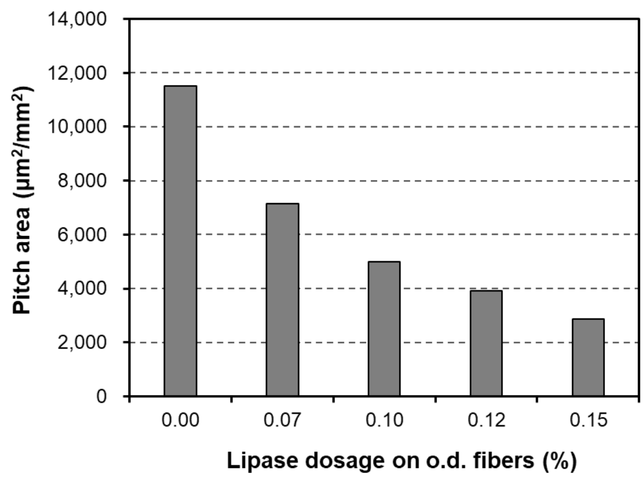

Fig. 9. Areas of stained pitch particles in OCC sheets depending on different lipase dosages

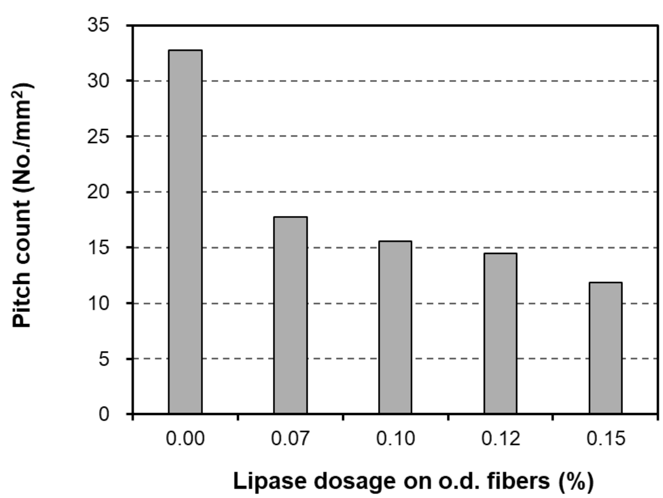

Fig. 10. Counts of stained pitch particles in OCC sheets depending on different lipase dosages

CONCLUSIONS

1. Lipase enzyme was able to effectively control pitch and/or stickies contamination in recycled pulps sourced from a duplex board mill. Sudan IV staining and image analysis confirmed substantially higher pitch-like contents in OB and OCC, which can be attributed to the presence of inks, adhesives, and coating agents. The amounts were higher than in OMG and ONP. Meanwhile, OMG exhibited the lowest pitch contamination among the recycled pulps.
2. Lipase activity was highest during the first 30 min. At this point, the reduction in stained area was most significant, and larger pitch aggregates fragmented into smaller pieces, resulting in a slight increase in the number of stained particles. These findings indicate that a reaction time of 30 min is adequate for lipase activity and suggest cost-effectiveness for industrial applications.
3. Lipase decreased the pitch or stickies content in both OMG, which had the lowest stainable content, and OCC, with the highest stainable content, but the pitch or stickies control efficiencies differed between the two pulp types. In OMG pulp, the enzyme activity was limited by ink-related compounds, whereas in OCC pulp containing enzyme-accessible lipids, lipase efficiently reduced the stainable content in a dose-dependent manner. This suggests that the wax components in OCC may have been degraded by lipase.

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