Advances in the Control of Dissolved and Colloidal Substances Present in Papermaking Processes: A Brief Review

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In the production of paper, especially when using mechanical pulp or recovered wood-containing paper, a large amount of dissolved and colloidal substances (DCS), mainly composed of hemicelluloses, extractives, lignans, and lignin-related substances, are released from the pulp and dissolved or dispersed into the process water. The accumulation of DCS during the papermaking operations due to the closure of process water systems gives rise to various detrimental impacts on the papermaking process and the resulting paper products. Thus it is indispensible to remove or control the DCS in order to overcome or alleviate their negative influences. This review emphasizes recent advances in control of DCS by physical, chemical, and biological methods. The widely used fixatives such as aluminum sulfate, poly-(aluminum chloride), polyamines, polyvinyl amine, and highly cationic starch, as well as their functional effectiveness, mechanism, influencing factors, and influences on paper products are considered. Simultaneously, biological treatments including fungal treatment and enzymatic treatments with lipase, pectinase, laccase, and immobilized enzymes, are also assessed in detail. DCS control has been an important way to improve the runnability of paper machines and the quality of wood-containing paper and recycled paper products. Advances in DCS control are likely to create additional benefits to the papermaking industry in the coming years.

Keywords: Mechanical pulp; White-water; Dissolved and colloidal substances; Chemical control; Biological control

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INTRODUCTION

With the rapid development of the papermaking industry, increased amounts of mechanical pulp and deinked pulp are being used in the production of various paper products. However, during pulping and bleaching operations, a large amount of dissolved and colloidal substances (DCS) are released from the wood material and are introduced into the process water system. Some of them that are negatively charged are also called "anionic trash" because some components of DCS released from wood commonly contain carboxylic acid groups that will dissociate to their negatively charged carboxylate forms under the conditions of pH typically associated with papermaking processes (Holmbom and Sundberg 2003; Hubbe *et al.* 2012). It has been reported that the dissolved substances are mainly hemicelluloses, pectins, lignans, lignin, and lignin derivatives, whereas the colloidal substances are mainly composed of lipophilic extrac-tives including fatty acids, resin acids, triglycerides, steryl esters, and sterols (Holmbom and Ekman 1991; Nurmi *et al.* 2004). Among these DCS components, the anionic hemicelluloses, pectic acids, oxidized lignin, anionic colloidal extractives, and sticky colloids are considered to be most detrimental to the papermaking process. Usually, the amount of dissolved substances, which comprises about 60% of DCS, and in some cases can make up 95% of the DCS, is more than that of colloidal substances (Holmbom 1997; Basta *et al*. 2003; Miao *et al*. 2007; Miao 2009). Some studies have also shown that the dissolved substances are largely responsible for the relatively high cationic demand, the chemical oxygen demand, and the conductivity of such pulp suspensions; nevertheless, the colloidal substances mainly contribute to the turbidity and the zeta potential (Nurmi and Eklund 2000; Wang 2003a; Liu 2008). The subjects of DCS and cationic demand were recently reviewed, with an emphasis on the different chemical components of DCS (Hubbe *et al.* 2012). The present review goes into greater detail with respect to control strategies for DCS, with a primary focus on chemical and biological measures.

Modern pulp and paper mills are being encouraged to minimize both water usage and effluent discharge, especially in highly integrated paper mills producing wood-containing paper grades, and so the progressive closing of paper mill water circulation is inevitably adopted. As a consequence, increasing amounts of dissolved and colloidal substances accumulate in the mill process water. It has been found that these DCS can give rise to various problems in the papermaking process and in the quality of the paper products. In particular, the small colloids and some anionic dissolved substances are the most troublesome. Problems include deposits on machine parts (Maher *et al*. 2007), decreased wet strength (Zhang *et al*. 1999), interference with cationic retention/drainage aids (Dunham *et al*. 2002), decreased sizing degree (Li *et al*. 2002), reduced sheet brightness (Zhang *et al*. 1999), and impaired paper strength (Lindström *et al*. 1977). The detailed disadvantages or limitations are as follows.

- The dissolved and colloidal substances can exist stably in pulp or white water based on electrostatic or steric mechanisms. When pulping and papermaking conditions are changed, mainly referring to pH value, temperature, shearing force, and addition of inorganic salts or organic polymers, these substances may be destabilized and deposit on equipment surfaces or fabrics, often causing the paper web to break, and this consequently reduces the paper machine operating efficiency (Sjöström 1990; Wågberg and Ödberg 1991; Zhang *et al*. 1999; Wang *et al*. 2003b).
- The efficiency of cationic paper chemicals that are added to improve the paper machine runnability and the paper quality will be decreased because some dissolved and colloidal substances have a negative electrostatic charge (Whipple and Maltesh 2002; Desharnais *et al*. 2002; Dunham *et al*. 2002; Zhang *et al*. 2007; Miao *et al*. 2010). Since these anionic substances will compete with fibers and microfibers, they will interfere with cationic additives such as drainage aids and retention aids, strengthening agents, and other additives to the process. As a result, the drainage time increases (Dunham *et al*. 2002), the retention of fines or fillers decreases (Miao 2009), and even the wet-web strength of paper decreases (Zhang *et al*. 1999).
- The dissolved and colloidal substances also have an adverse effect on the paper quality. It has been claimed that the strength properties and the brightness can be decreased because of the addition of DCS (Zhang *et al*. 1999). Meanwhile, the paper quality is not only impacted by the amount of DCS, but also affected by the

different chemical nature of the components and their physical form (Zhang *et al.* 1999; Hubbe *et al.* 2012). It has been discovered that the decrease of paper strength is attributable to the presence of colloidal substances, and the optical properties are mainly affected by the dissolved substances (Wearing *et al.* 1985; Zhang *et al.* 1999; Zhao *et al*. 2003; Chen 2004).

- Additionally, the different components of DCS also have various effects on the flotation of deinked pulp. More lipophilic extractive compounds which are added into the recycled fiber suspension can reduce the flotation efficiency, and the pectic acids and calcium ion can decrease the flotation yield (Yang *et al.* 2010a).

In order to overcome or alleviate the above disadvantages or negative effects induced by DCS, the amount of dissolved and colloidal substances present in the pulps or circulating in the white water systems have to be controlled or maintained at a constant level. It is believed that well-optimized treatment of DCS can be highly favorable to improve the paper machine runnability and the paper product performances.

Nowadays, strategies to effectively control or remove DCS and to overcome their harmful effects on paper production have become a hot topic. Plenty of methods can be used to treat the DCS, such as physical methods, chemical methods, and biological methods. Among these approaches, physical methods are usually used for removing the DCS present in white water; chemical methods principally use fixing agents to neutralize and destabilize part of the DCS, leading to the retention of some DCS components in the paper web and the removal from papermaking system. Biological methods have been much more frequently investigated and used in recent years. These are mainly applied to degrade some components of DCS, but the byproducts are not removed from pulp or white water. In the present paper, both chemical and biological methods are principally reviewed and discussed.

PHYSICAL STRATEGIES FOR REMOVAL OF DCS

Some physical methods that are used to treat white water may be effectively utilized to reduce the amounts of DCS materials present in process water. These physical methods below are mainly used to remove DCS partly or completely from white water, reducing the amount of DCS entering the papermaking system with the recirculation of white water. These methods include dissolved air flotation (Ben *et al*. 2003), evaporation (Stevenson 1990), disk screening and precipitation clarification (Farlow 1996; Ravnjak *et al*. 2003), and freezing crystallization (Kenny *et al*. 1992).

Dissolved air flotation (DAF) is a commonly used method to purify the process water in de-inking operations in newsprint mills. It can effectively remove suspended solid substances, but the efficiency of a DAF unit is very low in DCS removal (Ben *et al.* 2003). Nevertheless, it should be noted that the DAF can have a stable performance in removing DCS components, especially when combined with some flocculants or coagulants; such combined approaches have been shown to remove most of the colloidal components of DCS (Monte *et al.* 2011; Miranda *et al.* 2009 a,b; Saarimaa *et al*. 2006; Opedal *et al.* 2011) .

Evaporation has been utilized to treat white water or process water, and it has also enabled the complete closure of process water in a paper mill; however, it has not been widely used because of the high investment and operating cost. Successful removal of DCS by precipitation needs to consider two conditions: firstly, the concentration of DCS must be sufficiently high to ensure economical precipitation efficiency; secondly, the mixture of DCS and flocculants should be suitable for removal by precipitation. Crystallization by freezing can be employed to remove DCS effectively, and the key is to control the freezing speed. Nevertheless, it is very difficult in actual operation, and the investment is also relatively high.

Membrane technology is assumed to be an effective method for removing DCS present in white water, especially when combined with biotechnology. Such a combined approach can remove 100% of fatty and resin acids, 72 to 84% of dissolved chemical oxygen demand, and 18 to 37% of total dissolved substances (Paleologou *et al*. 1994; Elefsinioties 1997). However, its utilization is limited due to a relatively high cost, and the fact that the membrane pores are subject to occlusion.

Another important strategy is improved washing systems such as press washing (Bräuer *et al*. 2001). Nevertheless, due to the increased costs of water treatment and the fact that most substances being removed through press washing are the dissolved substances such as hemicelluloses and pectins, rather than the more detrimental colloidal substances, washing may cause other problematic situations.

Therefore, the physical methods tend to be only used as pretreatments or after-treatments for the removal of DCS. The efficiency of removing DCS may be improved greatly when physical methods are combined with other controlling technologies.

CHEMICAL STRATEGIES FOR REMOVAL OF DCS

Compared with other methods, chemical fixation has been more widely employed due to its convenience and relatively high efficiency in removing DCS from process water. Since the 1970s, there have been many articles recommending the use of fixatives to at least partly neutralize the excess DCS in paper machine systems in order to achieve more rapid drainage and higher retention (Auhorn and Melzer 1979; Linhart *et al.* 1987; Hubbe *et al.* 2012). The terms fixing agents or fixatives commonly refer to inorganic or organic polymers with relatively low molecular mass and high cationic charge density. Some anionic components of dissolved and colloidal substances can be fixed onto fibers or fillers and be removed from paper machine system together with the wet sheet, as it is being formed, which renders the paper machine system cleaner. Currently, fixatives available can be divided into three categories that include the inorganic aluminum compounds aluminum sulfate and poly-(aluminum chloride) (PAC) (Allen 1981); organic compounds such as polyamine (PA), poly-diallyldimethylammonium chloride (poly-DADMAC), poly-ethyleneimine, and poly-vinylamine (Tanaka and Ödberg 1992; Tanaka *et al.* 1993; Esser *et al.* 2005; Leduc and Mcallister 2005); and semi-synthetic compounds based on the natural high polymers, for example, highly cationic starch or highly cationic guar gum (Wang *et al*. 2006b; Wang and Xing 2006).

Characteristics of Different Fixatives

Different fixatives display different interaction characteristics towards pulp's chemical components because of their different molecular mass, charge density, and molecular configuration. Meanwhile, the synergistic effects between fixatives and retention/drainage aids are also diverse. The intended effect of a fixative is to remove DCS components from the system by increasing their deposition onto fibers, allowing them to be removed from the water system and to be retained in the paper web. The efficiency of controlling DCS with aluminum sulfate, which has been widely used for many years, depends greatly on the pH value of the system, as well as the concentration of the aluminum ions. The highest efficiency often can be obtained when the operating pH is adjusted to between 4 and 4.5 at 20 mg/L of aluminum ions concentration (Allen 1980). Thus, aluminum sulfate is suitable for removing DCS present in mechanical pulp that is used for the production of newspaper under acidic conditions. Nevertheless, the use of aluminum sulfate can bring about problems of pitch deposition and equipment corrosion. It has been shown that a combination of aluminum sulfate and sodium hydroxide can improve the fixing efficiency (Halvarson and Halt 1983). This is probably due to the formation of aluminum oligomers, the efficiency of which is better than that of aluminum sulfate when they are used to control DCS. However, under alkaline conditions the efficiency of removing DCS with either aluminum sulfate or poly-aluminum chloride is still lower than organic compounds due to their rapid decrease of charge density at neutral to alkaline pH.

Quaternary ammonium polyelectrolytes having linear molecular structure or branched chains and crosslinking structures have been the most pervasive class of fixative (Gill 1996). The molecular mass and the charge density are diverse because of different synthesis methods. Their charge density usually remains constant under neutral and alkaline conditions, which leads to more stable action (Zhou 2006). Poly-diallyl-dimethylammonium chloride is considered to be a non-toxic cationic poly-(quaternary ammonium) salt that can be available with different molecular mass. It has high charge neutralization capacity; when its molecular mass is relatively high it also has a certain bridging ability that can give rise to the flocculation of some anionic colloidal particles (Gill 1996). The use of poly-diallyldimethyl ammonium chloride is not as prevalent as that of other polyamines due to its higher price, though it has been claimed to be very effective under controlled conditions. Li *et al.* (2002) reported that the efficiency of internal sizing agents alkylketene dimer (AKD) or rosin ester sizing could be improved by the addition of the poly-(DADMAC) in the presence of DCS from BCTMP.

The effectiveness of polyethyleneimine with lower molecular mass is similar to that of polyamine when it is used to control DCS, and it can be applied in different kinds of pulps. It is noticeable that the cationic charge density of polyethyleneimine is affected by the pH value to a certain degree. Though the charge density will decrease under alkaline conditions, its efficiency of removing DCS is still higher than that of many other cationic polymers under neutral or slightly alkaline conditions. PEI was also found to be strongly adsorbed onto the fibers, leading to an increase of fixation of resin acids and triglycerides to fibers when it was added into the fiber suspension with the presence of DCS (Maher *et al*. 2007). PEI has been more widely used in newspaper production due to its higher removal efficiency of some DCS components.

Polyvinyl amines are another type of fixative, also finding use in treating DCS, especially for the control of hydrophobic contaminants such as latex from the repulping of coated broke (white pitch) (Leduc and Mcallister 2005). According to the structure, polyvinyl amine has an ideal macromolecular skeleton and good water-solubility. Its high cationic charge is expressed through amine radical chains, such that polyvinyl amine can be adsorbed effectively onto all kinds of natural or synthetic anionic surfaces (Fan and

Wang 2006). The effect of polyvinyl amine in treating DCS can be tuned by altering its molecular mass, cationic charge density, and hydrophobic groups (Esser *et al.* 2005). They can be flexibly utilized in removing the colloidal DCS components.

Just like poly-(diallyldimethylammonium chloride), the prices of polyvinyl amine and polyethyleneimine tend to be relatively high, which limits their more widespread utilization. Nowadays, highly substituted cationic starch, as a very important branch of fixatives, may be widely utilized in the future due to the convenient availability of the base material, its greener nature, and the lower price of raw materials. Such additives have the characteristics of sustainability, renewability, and biodegradability. In the literature (Vihervaara and Jouko 1994), when a highly substituted cationic starch, whose charge density was 2.8 meq/g, was used to treat the ground mechanical pulp, the ability of adsorbing anionic trash was obviously higher than that of poly-(diallyldimethyl-ammonium chloride). Under many circumstances, the amount of adsorbed anionic trash can exceed the theoretical calculation. Bobacka *et al*. (1999a,b) reported favorable effects when highly cationic starch, whose degree of substitution was 0.8, was applied to treat DCS present in hydrogen peroxide bleached thermomechanical pulp. If highly cationic starch with a substituted degree 0.405 was used to treat the white water, the chemical oxygen demand of white water could be substantially decreased (Yang *et al*. 2003). However, if the cationic starch without decrease of molecular mass was directly applied to treat waste pulp, then the effect was not as good as that of polyamine fixatives (Wang *et al*. 2006). So the removal efficiency of DCS with cationic starch is closely related to its molecular mass.

Fixation Mechanisms

The mechanism of fixation is rather complicated, but there are still some general ideas of how the fixation process occurs. The fixation mechanism can be understood in terms of two stages. The first stage appears to involve the precipitation of dissolved substances, combined with flocculation of colloidal particles, leading to the formation of polyelectrolyte complexes with the fixatives (Wågberg and Ödberg 1991); in the second stage, these formed complexes can be removed by filtration in the paper sheet or by adsorption on fiber surfaces, leading to removal from the white water system as part of the paper web. If the total surface charge on the formed complexes is neutral, their removal may occur mainly by filtration; however, if the overall surface charge is positive, adsorption on fiber can take place (Esser and Auhorn 2001). It is generally considered that the basic theories of cationic polyelectrolytes interacting with dissolved and colloidal substances are mainly based on electrostatic charge neutralization, patching coagulation, and bridging flocculation (Shetty *et al.* 1994; Chen and Chen 2008; Wang *et al*. 2008). Charge neutralization is a process in which polyelectrolyte complexes will form due to the mutual adsorption of anionic and cationic ions under the electrostatic attraction (Chen and He 2000). Two idealized patterns of complexation have been proposed, *i.e*. a ladder-shaped structure and an ellipsoid egg structure (Michaels 1965). The compounds can precipitate from the solution when the ratio of anionic and cationic ions approaches an ideal value of 1; otherwise, the generated materials can only induce the solution to be cloudy (Brouwer 1991; Bley 1992; Chen and He 2000). Partial anionic charge of a particle will be neutralized or even be reversed to be cationic after the addition of fixatives, which causes the mutual attraction of particles with opposite charges, and then these particles aggregate based on the patching coagulation principle. The adsorption process between particles and polyelectrolytes is not reversible, but the formed aggregates are very sensitive to the shearing force (Eklund 1991). These aggregates can be dispersed under the strong shearing force, and a considerable amount of particles will go back to their initial status under the influence of charge attraction after the removal of shearing force.

In the case of cationic polymers with higher molecular mass, their molecular chains can extend fully in water due to the mutual repulsion between charged groups. Afterwards, the extended molecular chains come into contact with the particle surfaces of contaminants, and segments of the molecules are adsorbed onto the particle surfaces. Other molecular chains may be favorably adsorbed onto fibers or another particle. This is usually called bridging. However, a majority of fixatives are those polyelectrolytes with molecular mass below one million g/mole; thus it is believed that bridging flocculation plays only a minor role in the removal of DCS in typical cases. Sometimes the combination of fixatives and traditional retention chemicals will be inevitably involved with the flocculation mechanism (Rebarber 1995; Wang *et al*. 2005).

Besides the above mentioned mechanisms, for highly cationic starch and other special fixatives, hydrogen bonding can play an important role in controlling DCS. In the literature, the same efficiency as other fixatives could be obtained when highly cationic starch was used, although its charge density was lower than other cationic polyelectrolytes (Vihervaara *et al.* 1990). There are two possible reasons that explain such effectiveness of highly cationic starch. On the one hand, the fixation mechanism of highly cationic starch is likely to be based on the flocculation, because the paper uniformity may be worse than that achieved with the use of other fixatives (Wang *et al*. 2006). On the other hand, the particular polyhydric cyclic molecular structure leads to the easy formation of hydrogen bonds between cationic starch and other polysaccharides, and the latter will be removed. The study of several highly cationic guar gums with different molecular mass indicated that guar gums with higher molecular mass could be more easily adsorbed by fibers; nevertheless, the guar gums with lower molecular mass could interact much more readily with DCS (Wang and Xing 2006). These authors reported that though the charge density of the guar gum derivative was far lower than that of polyamine, the effectiveness in removing colloidal substances and chemical oxygen demand was the same or better than that of polyamine, which indicates that the fixation mechanism of highly cationic guar gum also involves hydrogen bonding. Nano-size TiO_2 can also be used to remove DCS by hydrogen bonds that form between the surface hydroxyl (Ti^{4+}-OH) and the functional groups containing an oxygen atom of DCS; the dissolved substances can be captured, and the colloidal substances and complexes can be bridged by the nano-size TiO_2 particles, inducing agglomerate flocculation (Chen *et al.* 2011). Additionally, the dual-component systems with retention aids composed of TiO_2, nano-TiO_2/APAM, and nano-TiO_2/AmS, can be utilized and successful for the removal of DCS in deinked pulp (Chen *et al*. 2004, 2006, 2009; Kou *et al*. 2008).

Research (Lin and Xu 2002) has reported that sticky substances can be fixed on fibers by aluminum sulfate, but they would easily turn into larger particles, and this would have an adverse impact on the paper appearance; on the contrary, some high molecular cationic fixatives would disperse and fix these small particles onto the fibers. The research of Lee *et al.* (2005) demonstrated that it was easier for fixatives to remove colloidal substances than to remove dissolved substances; for example, the resin components in coated waste paper can be more easily removed by fixatives than those in the mechanical pulp, and this is because the main contaminants of coated waster paper are latex materials. Especially, some dissolved substances such as some degraded polysaccharides are much more difficult to be removed by fixatives. Frequently-used fixatives can only decrease the chemical oxygen demand, which generally represents the relative content of dissolved substances of a highly polluted system by 100 to 400 mg/L (Gill 1996). Not all the fixatives have the ability to decrease the chemical oxygen demand dramatically. This is possibly because the relatively weak anionic nature of some dissolved substances, or their lower molecular mass, gives rise to difficulty in the formation of complexes between the fixatives and the dissolved anionic species. These experimental results show that the removal of colloidal substances is based on the coagulation mechanism. When a combination of fixatives and retention aids is adopted, the efficiency of retention aids will be increased due to the neutralization of anionic trash, and the colloidal substances can also be effectively removed from the system based on the bridging mechanism of the retention aids.

Theories to account for fixation include charge neutralization, patching coagulation, bridging flocculation, and even hydrogen bonding. It is indispensible to consider the charge density, molecular configuration, molecular mass and its distribution, and the acting patterns between fixatives and DCS to interpret reasonably the action characteristics of fixatives. It is also very important to analyze and determine the composition, the content, and the deposition nature of some DCS components in order to further clarify the fixing mechanism of different fixatives. A large amount of literature can be referred to for help in analyzing the DCS components (Douek 1997; Yang and Chen 2003; Wang *et al*. 2003; Hubbe *et al.* 2012).

Factors Influencing the Action of Fixatives

Based on a charge neutralization mechanism one can assume that when the charge density of fixatives is higher, the fixation ought to be more effective. Part of the cationic charge is neutralized by the dissolved anionic trash, and the rest of the cationic charge is used to react with the colloidal substances based on the coagulation mechanism. The higher of the charge density of fixatives, the stronger will be the coagulation. A study (Tian 2005) showed that the resin deposition problem can become more serious if the fixatives charge is not enough to fix the resin onto the fibers. An optimal dosage of fixatives should be chosen; otherwise, the expected result will not be obtained. Only at steady state would the addition level of fixatives be expected to be in line with the amount of DCS present in the pulp slurry within a specified period (Hubbe *et al*. 2012). Also, there can be a waste of chemical if the dosage is significantly higher than the point of neutralization, even causing the redispersion of DCS. The system charge is inevitably reversed if the fixatives are added in excess, and this brings about an unstable wet-end system that influences the runnability of the paper machine and the quality of the paper.

Many studies have indicated that whereas the charge neutralization and coagulation mechanisms are important, the effects of molecular mass and molecular configuration of the fixatives can have a greater influence on the fixing efficiency in some cases (Gill 1993; Ravnjak *et al.* 2003). Typically the fixing efficiency increases with increasing molecular mass of a given type of fixative. If a fixative with fixed molecular mass cannot fix the resin and stickies on the fibers, then such materials are likely to become concentrated in the white water system, finally leading to deposit problems (Tian 2005). Concerning the molecular configuration, better results sometimes have been obtained when fixatives with more side-chains or cross-linked points are used (Tian 2005). Moreover, the molecular configuration of fixatives presumably determines the compatibility between the fixatives and the pulps, which can be more important than whether the charge density is at its optimum level.

The location in a paper machine system where the fixatives are added plays an important role in removing DCS. Generally when the fixatives are added into high consistency pulp, they can contact more easily with DCS and fibers, resulting in the optimum fixing efficiency. But the reaction time is also very significant. Polymers with relatively short chains will permeate into the fibers and lose their fixing function when the time of contact with the fibers is too long. However, good results perhaps also cannot be obtained when the point of addition is too close to the headbox due to insufficient mixing or due to the shorter time of fixatives reacting with DCS components. The control of fixatives addition can also be combined with online charge titration equipment, which is capable of automatically sampling filtrate from the paper machine system (Rice and Roeraade 2003; Hubbe *et al.* 2012).

The fixation effectiveness also depends on the pH value, conductivity, and the temperature of pulp or whitewater. For example, the cationic charge density of aluminum sulfate, poly-(aluminum chloride), or polyethyleneimine will decrease as the pH value increases in the range of about 5 to 8. In addition, alkaline pulping will also generate more alkaline hydrolyzed products such as alkaline lignin and pectic acids, which yield a more anionic system (Holmbom and Ekman 1991; Thornton *et al.* 1993; Pranovich *et al.* 2003; Thornton 1994; Sundberg *et al.* 1998). Under such conditions the fixatives dosage must be increased in order to obtain a better fixing effect (Brouillette *et al.* 2005). Also, an increase of pulp temperature can give rise to the release of more anionic materials from the fiber stock, which will decrease the fixatives' effectiveness. When a polyamine is used to treat white water, though the increase of conductivity could deteriorate the performance of the polyamine, better efficiency could still be obtained under the higher conductivity conditions (Wang 2011).

Besides the above-mentioned influencing factors, retention aids (Rebarber 1995; Wang 2005) and selective adsorption onto different pulp components will further affect the effectiveness of fixatives. An effective retention aid system can be critical to the performance of fixatives and other wet-end additives. In the absence of an effective retention aid system, there can be adverse effects on drainage rates when DCS forms precipitates with coagulants or flocculants (Hubbe 2008; Hubbe *et al*. 2009). Such precipitates can play a role similar to that of cellulosic fines, having a size range which tends to plug up the drainage channels within a wet paper web. An effective retention aid system can prevent hold such fine matter onto fiber surfaces, thus overcoming the plugging effect. The selective adsorption of fixatives onto different solids in pulp suspension is also very important for the fixing efficiency. Polyethyleneimine will be preferentially adsorbed onto fibers and fillers when its dosage exceeds certain level, but the polyamine tends to be adsorbed onto DCS prior to other components (Wang 2005).

Effect of Fixation on the Paper Properties

When DCS is fixed on the fibers due to the addition of fixatives, the incorporation of the DCS in the sheet will give rise to various effects on paper properties. This is a matter of concern for paper machine managers, researchers, and customers. The physical properties of paper are affected not only by the total amount of DCS, but also by their intrinsic characteristics. A study indicated that the strength of paper prepared with 90% of TMP and 10% of ONP was mainly impacted by the colloidal substances (Francis and Ouchi 2001; Rundlöf *et al.* 2002; Tay 2001), but another study showed that the strength of paper prepared with TMP was mainly affected by the dissolved substances (Zhang 2000b). Tay (2001) pointed out that the decrease of paper strength was mainly caused by the hydrophobic materials that were fixed on the fibers, hampering the inter-fiber bonding of CTMP. As to the impact of metal ions on paper strength, Springer *et al.* (1986) found that the iron ion had the greatest effect in comparison with aluminum, and that the sodium and calcium ions had the least effect.

However, results obtained with mechanical pulp might be the same as those for recycled pulp because of their different DCS constituents. The DCS components present in waste pulp can be mainly hot melts, pressure sensitive adhesives, waxes, synthetic toner ink binders, and latex coating ingredients (Liu 2006). These materials are sticky and belong to the category of microstickies. When they are fixed on the fibers in a stable state, they can be expected to improve the paper strength through paper formation and drying, and this strengthening action may be similar to the addition of cationic latex into the wet-end of paper machine (Alince 1999). Wang *et al*. (2006b) reported that the decrease of folding resistance of paper was the most notable when the microstickies were fixed on the fibers; the inorganic poly-(aluminum chloride) in itself could bring about a negative effect on the tearing index and the tensile index; but an organic polyamine, poly-(diallyldimethylammonium chloride), and a highly cationic starch did not decrease either strength property. Depending on conditions of hydrodynamic shear, the undegraded highly cationic starch with high molecular mass may adversely affect the paper's uniformity due to the high levels of flocculation.

Additionally, it is an interesting topic to consider whether the fixatives themselves have a strengthening function. For instance, cationic starch with a lower degree of substitution is commonly used to increase paper's dry strength, whereas cationic starch with higher substitute degree can be used for the coagulation. It follows that when strength and drainage are both considered, cationic starch with medium degree of substitution can be chosen (Shen 2001). Only part of the cationic charge is consumed when highly cationic starch interacts with colloidal substances, and the excess of positive charge on the treated colloids allows them to be adsorbed onto the fibers. It is worthy of research to find out whether the coagulated substances have a strengthening function. Several types of highly cationic guar gums with different molecular mass have been studied, and the results indicated that the highly cationic guar gum could enhance the strength of pure chemical pulp, especially for the strength of folders (Vihervaara *et al*. 1990). Though the strengthening function was weakened when the highly cationic guar gum was used to treat the recycled pulp, the increase of folder strength was still obvious, and the tensile index and the tearing index remained unchanged.

CHEMICAL STRATEGIES FOR OVERCOMING DCS DETRIMENTAL EFFECT

Chemical methods have significant potential for controlling DCS. Such methods mainly involve dispersive stabilization, adsorption, surface passivation, and oxidation (Wang *et al*. 2006a). Dispersive stabilization mainly employs surfactants or dispersants to stabilize the colloidal substances, thus reducing their tendency to coagulate or to form deposits (Allen 1981, 1982). Some polyelectrolytes with high cationic charge density and low molecular mass are diluted and sprayed onto the fabrics or wires and other paper machine surfaces; this operation leads to the increase in the hydrophilic nature of these surfaces, resulting in a decrease of the colloidal DCS components deposition; this process is usually termed surface passivation (Tanaka *et al.* 1993; Duy 1998; Esser *et al.* 2005).

Adsorption processes employ talc, bentonite, or synthetic fibers to collect colloidal particles of DCS and to decrease their tackiness (Tanaka and Ödberg 1992; Sithole 1992). Cationic talc has been used to treat deinked pulp; it was shown that the DCS could be reduced from 1989 mg/L to 1927 mg/L at an addition level of 2% cationic talc (Gao 2011); such an effect might be explained by a change in the charge balance in the pulp system, resulting in the loss of stability of some DCS, allowing it to be easily agglomerated with each other and finally become trapped by talc particles (Gao *et al.* 2011). Tijero *et al.* (2012) also used talc to treat furnish in different paper machines and observed a 25% to 50% reduction in the cationic demand of process water compared to a control condition without talc addition. A dual-component system consisting of a cationic-modified microporous zeolite and an anionic polyacrylamide-based polymer was used to adsorb hydrophobic and anionic aromatic DCS (Huang *et al*. 2006). But this approach based on adsorption onto mineral surfaces may result in deposits and paper brightness reduction, so its use is limited.

Oxidation treatments have also been tested for their potential to degrade organic contaminant from white waters. Ozone has been utilized in mechanical pulp waste waters to selectively destroy resin and fatty acids (Roy-Arcand 1996; Tay 2001). However, the high cost of ozone generation and operation limits its commercial application. Among these methods mentioned above, the adsorption is more widely used, especially the application of talc in newspaper mills.

BIOLOGICAL STRATEGIES FOR OVERCOMING DCS DETRIMENTAL EFFECT

Biotechnology has been extensively used in different aspects of pulp and paper manufacturing including bleaching (Camarero *et al*. 2004), refining (Gil *et al*. 2009), deinking (Bajpai and Bajpai 1998), pitch control (Gutiérrez *et al*. 2001), fiber modification (Liu *et al*. 2009), and effluent treatment (Pokhrel and Viraraghavan 2004). Due to the disadvantages brought by DCS, many methods have been employed to solve these problems. One possible way of coping with the detrimental impact of DCS has been by chemical modification of them, and some problems, especially the pitch-deposition, induced by DCS can be efficiently resolved. Meanwhile, biotechnology is also another important approach which could be successfully used to alleviate the adverse impact of DCS. Because of the specific nature of DCS materials, enzymes have significant potential for selectively modifying polysaccharides, extractives, and lignin. The addition points are also very flexible, and the fungus/enzymes can be supplied to treat wood chips, fibers after screw press, fibers after refining, pulp products, and white water. However, whether the targets are fibers or white waters, the DCS in themselves probably will not be removed completely from papermaking system. Rather the application of biotechnology may be used to partly or completely degrade DCS and alleviate their detrimental impact.

Fungal Treatment of DCS

As shown from studies of mechanical pulp and newsprint mills, the dissolved and colloidal substances present in the pulp and white water are mainly derived from the woody components (Sjöström 1990). Treatment of the process waters or pulps using selected fungal species has been shown not only to degrade detrimental substances, but also to bring the additional benefit of a pulp yield increase (Eriksson *et al.* 1993). Lindberg *et al*. (2001) tried nine bacteria isolated from a paper mill in a laboratory to control the DCS from thermomechanical pulp. The results indicated that the bacteria could hydrolyze triglycerides to free fatty acids, and the liberated unsaturated fatty acids were also degraded to some extent, but the saturated fatty acids were not notably degraded. It seemed that the degree of unsaturation was of greater importance for the degradation of fatty acids than the molecular mass. About 30% of the steryl esters were degraded during 11 days; approximately 25% of the dehydroabietic, and 45% of the abietic and isopimaric resin acids were degraded; no degradation of dissolved hemicelluloses could be observed. However, the long reaction time and the limited temperature range for effective action of the fungi can render a direct microbial treatment impractical under the mill conditions. Compared with fungal treatment, cell-free enzymes have the advantages of shorter reaction time and a greater tolerance to higher temperature, although the pH conditions in paper machine systems limit a wider application (Hubbe *et al.* 2012). According to the diverse chemical nature of the different DCS components, a variety of different enzymes are needed to control DCS.

The use of enzymes in the pulp and paper industry has led to a great development in the last two decades. The enzymes that are widely studied and utilized in controlling DCS are mainly esterases, lipases, pectinases, and laccases. Besides, other enzymes such as mannanases and cellulase can also be used to control DCS, but reports about them are very few. Earlier work on enzymatic treatments of DCS is shown in Table 1, which is based on the summaries by Buchert *et al*. (1997) and Rundlöf *et al.* (2002).

Table 1. Summary of Published Material about Enzymatic Treatment of DCS

Enzyme	Acting on	Effect
Lipase, Resinase A (Novo)	Extractives	Pitch control in a groundwood based paper mill
Esterase, *Aspergillus oryzae*	Acetyl groups in galactoglucomannan	Precipitation of galactoglucomannan
Pectinase (Ultra SP-L)	Polygalacturonic acids	Decrease in cationic demand
Cellulase, Pergalase A 40	Cellulose	Decrease in turbidity of TMP filtrates
Mannanase, Megazyme, *Aspergillus niger*	Glucomannan	Destabilisation of colloidal extractives
Xylanase, *Tricoderma reesei*	Xylan	Destabilisation of colloidal extractives

Lipase Treatment of DCS

Because the enzyme is water-soluble and the resin is hydrophobic, lipase treatment of DCS mainly acts on the colloidal particle interfaces and alleviates pitch deposition. It has been found that colloidal substances such as triglycerides and steryl esters are the dominant factors that can induce sticky deposit problems. Enzymatic treatment plays an important role in solving such problems. Mill experiences of the use of lipase indicated that it could be used successfully to control DCS (Fujita *et al*. 1992; Chen *et al.* 2001). The turbidity, cationic demand, and particle size of DCS from TMP were decreased to different degrees by lipase treatment (Wang 2007). A study reported that the lipase treatment of white water could deplete the triglycerides and part of the steryl esters, resulting in an increase of fatty and resin acids (Zhang *et al*. 2000b). Rundlöf (2002) also studied the effect of lipase treatment of model TMP dissolved and colloidal substances on handsheet properties; the results showed that the treatment of DCS with lipase gave a complete hydrolyzation of triglycerides into free fatty acids and gave higher fatty acids content and improved tensile strength on the same level as the reference. Other studies also indicated that the lipase treatment could increase paper strength (Mustranta *et al.* 1995, 2001; Buchert *et al.* 2002; Wang 2007). But the degradation of triglycerides could contribute to the cationic demand of DCS water samples.

Pectinase Treatment of DCS

The presence of pectins as fiber wall constituents in wood was reported for the first time in the beginning of the 20^{th} century (Hagglund 1928). Pectins in wood are concentrated in the primary wall and middle lamella (Westermark *et al.* 1986), and are largely composed of a backbone of methylated D-galacturonic acid units occasionally interrupted by L-rhamnose units. Mechanical and recycled pulps are usually bleached by hydrogen peroxide under alkaline conditions. The pectins can be degraded to low-molecular-mass fractions and demethylated during peroxide bleaching at high pH, which causes 1/3 of the original pectins in fibers to dissolve into process water as high-charge-density pectic acids (Thornton *et al.* 1993; Pranovich *et al.* 2003). The pectic acids are composed mainly of anionic galacturonic acid groups and constitute the major part of anionic charge (cationic demand) of bleached mechanical pulp water (Thornton *et al*. 1993). They may also consume cationic paper chemicals and are commonly regarded as detrimental substances, or so-called anionic trash (Holmbom 1991; Thornton *et al*. 1993; Kekkonen *et al.* 2002; Ricard *et al*. 2005a,b). Some studies have also shown that peroxide bleaching of mechanical or recycled pulps can lead to the drastic release of pectic acids and the increase of cationic demand of DCS in the water (Holmbom and Ekman 1991; Thornton *et al.* 1993; Pranovich *et al.* 2003; Thornton 1994; Sundberg *et al.* 1998; Miao 2007).

The use of pectinase to break down the polymeric pectic acids into monomeric galacturonic acid present in mechanical and recycled pulp or process water has been pursued by several research groups, and the approach has been used in some paper mills to alleviate the detrimental effects of pectins (Thornton 1993, 1994; Thornton *et al.* 1996; Reid and Ricard 2000; Ricard *et al*. 2005a,b). Enzymatic degradation of pectic acids results in a considerable decrease in the cationic demand for the depolymerization of pectic acids (Miao 2009; Zhao *et al.* 2009). Also, different kinds of pectinases have different effects on the DCS (Zhang 2010), and this is assumed to be caused by the different chemical structure and characteristics of pectinases. It is striking that the combination of pectinase and chemicals to treat aspen BCTMP can further decrease the cationic demand of DCS water compared to the sample only treated with pectinase (Yang 2010).

Additionally, pectinase treatment of DCS can improve drainage and retention efficiency at the wet-end, as well as improving paper quality. Li *et al*. (2006) used pectinase to treat the DCS present in BCTMP and found that the pectinase treatment could increase the retention of fines. Miao *et al*. (2010) has also applied pectinase to treat the DCS from bleached old newspaper deinked pulp, and the results showed that the drainage rate could be increased by 30%, and the breaking length and the burst index of paper increased by 33.3% and 16.7%, respectively. When the DCS present in aspen alkaline peroxide mechanical pulp was treated by pectinase, the treatment also increased the fines retention and the drainage rate in the presence of CPAM retention aid and poly-DADMAC charge-control agent (Miao 2009).

Laccase Treatment of DCS

Laccases are oxidative enzymes that act mainly on phenolic compounds. But the use of the laccase-mediated systems has greatly expanded the potential of laccases for the degradation of phenolic compounds (Bourbonnais and Paice 1990). Laccase/mediator systems have been widely investigated for pulp delignification, deinking, and bleaching (Call and Mücke 1997; Bajpai 1999; Xu *et al*. 2006). Recently a few researchers have also focused on their degradation of lipophilic extractives. Gutiérrez *et al*. (2006) used a high-redox-potential laccase from the basidiomycete *Pycnoporus cinnabarinus* in the presence of 1-hydroxybenzotriazole as a redox mediator to treat three different pulps, and the results revealed that the laccase-mediator treatment completely or partly degraded most of the lipophilic compounds including: (1) free and conjugated sitosterol in eucalypt paper pulp; (2) resin acids, sterol esters, and triglycerides in spruce pulp; and (3) sterols and fatty alcohols in the flax pulp. Molina *et al*. (2008) also reported that the laccase alone could only decrease the concentration of some unsaturated lipids; however, the most extensive lipid modification was obtained with the laccase-mediator system. From the cited studies it can be concluded that laccase or a laccase/mediator system can be effective when applied to treat DCS samples. The research conducted by Zhang (2000b) showed that the laccase treatment of white water had little effect on the resin and fatty acids of DCS; however, there was a marked decrease in the other lipophilic extractives and lignans, as well as an increase in lignin content due to the polymerization of the low-molecular-mass phenolic lignans. When the laccase/violuric acid system was used to treat the DCS present in aspen BCTMP, the decrease of cationic demand, turbidity, and lignin content could be observed compared with control sample, and the total amount of fatty acids decreased by 33% (Miao *et al.* 2011). It is apparent that the use of laccase or laccase/mediator to treat DCS is feasible and effective, especially for some lignin-derivatives and unsaturated extractives.

Combination of Fungal and Enzymatic Treatment of DCS

Combination of fungal and enzyme to treat DCS may bring about better results. Zhang (2002) reported a combined fungal and enzyme system for use in a TMP/newsprint mill with a closed water system to degrade various dissolved and colloidal substances present in a typical mill white water (MWW) obtained from a TMP/newsprint mill, a model recycled white water (RWW), and a membrane-filtered model recycled white water (MFWW). It was shown that the growth of the white-rot fungus *Trametes versicolor* on these waters resulted in significant reductions in the amount of total dissolved and colloidal substances. Different enzymes including laccases, cellulases, and lipases were produced and released into the white water during the growth of the fungus. These enzymes, which could be circulated in the white water, would be able to directly degrade the different DCS components. Fungal enzyme treatments of MWW and RWW broke down considerable amounts of the detrimental DCS components. The enzymes present in the fungal culture filtrate could degrade more than

90% of the lignans and ester-bonded extractives. The resin and fatty acids content were decreased by approximately 40% and 60% in the respective mill white water and model white water samples. The fungal and enzyme treatments also resulted in the polymerization of low-molecular-mass phenolics into higher-molecular-mass lignin types of material. It can be found that, under control conditions, the efficiency is higher using combination of fungal and enzyme to degrade some DCS components than using a single enzyme.

Other Enzymes Treatment of DCS

Besides the above-mentioned enzymes, other enzymes can also be utilized to treat DCS. Rundlöf (2002) studied the effect of mannanase treatment of model TMP dissolved and colloidal substances on the paper properties, and the results indicated that mannanase treatment gave rise to a decrease in strength compared with the reference at the same amount of extractives in the sheet. Moreover, the combined treatment with mannanase and lipase led to a more pronounced decrease in tensile strength; this might be because the destabilization of colloidal substances induced by the decomposition of galactoglucomannans due to the mannanase affects the distribution of the colloidal extractives in the sheet, making it more detrimental to paper sheet strength compared with the stable colloid.

Immobilization of Enzymes to Treat DCS

Due to the high price of liberated enzymes and an inability to recover them from reaction systems, their extensive use has been limited to a certain degree (Katehalski-Katzir 1993). In order to solve this bottleneck, the technique of enzyme immobilization has emerged since 1916 (Zhang and Yang 2000). Enzymes are combined with various carriers by different methods and exist in the substrate phase without dissolution; then the enzymes are concentrated or limited into a definite area, where they can react with the substrates. This process is called enzyme immobilization. Advantages of using immobilized enzymes compared with using liberated enzymes can include repeated reaction, maintenance of activity for a longer time, improved enzyme stability, enhanced efficiency, and lower total cost (Yesim 2005; López-Gallego *et al.* 2005).

In recent years, studies on the treatment of DCS with immobilized enzymes have rarely been conducted or reported. Liu (2010, 2012a) used modified chitosan to immobilize pectinase, which then was applied to treat the polygalacturonic acid (PGA) and white water. The results indicated that the effect of catalytic decomposition of PGA by immobilized pectinase was very significant; after 20 min reaction under the optimal conditions, the cationic demand of PGA was reduced by 87% and the cationic demand of white water could be reduced by 37% even while undergoing reuse for five times. Immobilized lipase was also applied to treat the white water (Liu 2011, 2012b). These authors showed that the degrading efficiency of resin deposits reached 66.8%, the average particle size of resin deposits decreased from 552 μm to 276 μm, and the turbidity decreased from 101 NTU to 80.8 NTU. Finally, it was found that the immobilized double-enzyme was very effective; the degrading efficiency of resin deposits was 74.3%, the average particle size of resin deposits decreased from 552 μm to 151 μm, the turbidity decreased from 101 NTU to 75.1 NTU, and the cationic demand decreased from 4.29 meq/L to 1.81 meq/L. It is believed that the technology of enzyme immobilization for controlling DCS has great potential.

Other Biological Treatment of DCS

Besides the utilization of fungal treatments and enzymes, other biological methods such as membrane biological reactor (MBR) and aerobic and anaerobic biodegradation have also been explored in control of DCS. Hall and his colleagues (1996) made use of MBR technology to treat white water, achieving 100% degradation of resin and fatty acids, 84% of dissolved organic material, and 37% of dissolved solids. Aerobic and anaerobic biodegradability of DCS in recycled water through laboratory simulation was also researched (Liang *et al.* 2010). The results showed that the aerobic disposal ratio of chemical oxygen demand from DCS could be increased by 15.3% by the addition of nutrient substances, and this could also promote degradation of lignin-related components of DCS. Interestingly, DCS was removed to an obvious extent by anaerobic microbes; the removal percentage of DCS and UV_{280} were more than 40.5% and 68%, respectively after one day of treatment, after which the degradation of DCS came to a standstill. These cited studies indicate that some technologies used for treatment of waste waters can also be applied to treat white waters for controlling DCS.

CONCLUSIONS

Large amounts of dissolved and colloidal substances are produced during pulping and bleaching processes, and these materials tend to build up within the recirculation loops of white-water systems. These substances have been shown to induce detrimental impacts on papermaking machinery and greatly reduce product quality. Hence, it is desirable to develop technologies to remove detrimental substances or alleviate their negative effects and enhance the closure of a mill's water loops. Among the numerous technologies, chemical and biological treatments can be regarded as effective strategies to alleviate the detrimental effects of DCS.

Nowadays fixation is the predominant chemical method and is widely used for the removal of DCS. The different chemical characteristics of various fixatives, such as molecular mass, charge density, and molecular structure, can lead to different interactions between fixatives and DCS. Furthermore, the system pH value and temperature, the reaction time, the cooperation between fixatives and retention aids, and the different composition of DCS originating from various raw materials can also greatly affect the fixatives' actions. It is worth further studying of the cooperation and the mechanism between fixatives and retention aids, the mechanism of removing DCS by hydrogen bond function, and whether starch or guar gum products can play a dual role as fixatives and strengthening agents. Though fixatives have been extensively utilized, the industry still has a long way to go in the implementation of automatic control of charge demand.

Biological treatments, mainly employing enzymes such as pectinase, lipase, and laccase, have been extensively studied and employed in academic institutes and papermaking industries as the second most important manner of DCS control. Biotechnology is attractive in that such approaches can deal with vast amounts of pulps or waters, resulting in the degradation of organic components of DCS and the improvement of paper quality. Although the effectiveness of enzymatic treatment is apparent, the high price and the vulnerability to variable conditions such as pH and temperature impose restrictions on their extensive application. Therefore, further research is recommended to focus on developing a biotechnology that can improve the adaptability of enzymes and extend the duration of enzyme activity, for example by enzyme immobilization.

Though each method has its own advantages when used to control DCS, the detrimental problems brought by DCS during papermaking processes will not be completely solved by using just one kind of technique. Physical methods, chemical methods, and biological methods must be effectively combined to control DCS in order to obtain a better runnability of paper machines and paper properties. It would be the best that the "detrimental DCS" could be removed or degraded from furnish and white waters, while the "favorable DCS" could be kept; how to reach this objective deserves future study.

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