

GELAGAR

JURNAL TEKNIK

- ⊙ APPLICATION OF DECOUPLE APPROACH FOR HARMONIC POWER FLOW CALCULATION
- ⊙ BENEFITS OF A CHELATING STAGE PRIOR TO PEROXIDE BLEACHING ✓
- ⊙ THE OPTIMAL SELECTION OF ELECTRIC PROPULSION MOTORS BY USING ANALYTIC HIERARCHY PROCESS (AHP)
- ⊙ A STATIC AND DYNAMIC TECHNIQUE CONTINGENCY RANKING ANALYSIS IN VOLTAGE STABILITY ASSESSMENT
- ⊙ A MODEL OF PRODUCT MANUFACTURING LEAD TIME IN A NON REPETITIVE MAKE TO ORDER MANUFACTURING SYSTEM
- ⊙ MODELLING AND OPTIMISATION OF REFINERY GASOLINE BLENDING
- ⊙ THE GAS-LIQUID SEPARATION BY VERTICAL CYLINDER CYCLONE SEPARATOR
- ⊙ VARIETY OF HOUSE FUNCTION IN NILASARI HOUSING OF GONILAN, KARTASURA
- ⊙ THERMAL CHARACTERISTICS OF RUSUNAWA - COKRODIRJAN D.I. YOGYAKARTA
- ⊙ DUCTILITY BEHAVIOR OF REINFORCED CONCRETE COUPLING BEAMS WITH DIAGONAL REINFORCEMENT BETWEEN DEFORM TYPE WITH CRT BAR TYPE



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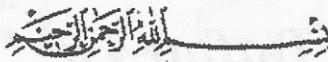
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Daftar Isi

	Halaman
DAFTAR ISI	i
PRAKATA	ii
APPLICATION OF DECOUPLE APPROACH FOR HARMONIC POWER FLOW CALCULATION	
<i>Agus Ulinuha</i>	83 - 90
BENEFITS OF A CHELATING STAGE PRIOR TO PEROXIDE BLEACHING	
<i>A.M. Fuadi, Harald Brelid</i> ✓	91 - 97
THE OPTIMAL SELECTION OF ELECTRIC PROPULSION MOTORS BY USING ANALYTIC HIERARCHY PROCESS (AHP)	
<i>Hadi Suroso</i>	98 - 105
A STATIC AND DYNAMIC TECHNIQUE CONTINGENCY RANGKING ANALYSIS IN VOLTAGE STABILITY ASSESSMENT	
<i>Muhammad Nizam</i>	106 - 114
A MODEL OF PRODUCT MANUFACTURING LEAD-TIME IN A NON REPETITIVE MAKE TO ORDER MANUFACTURING SYSTEM	
<i>Nur Indrianti, Isa Setiasyah</i>	115 - 124
MODELLING AND OPTIMISATION OF REFINERY GASOLINE BLENDING	
<i>Rois Fatoni</i>	125 - 133
THE GAS-LIQUID SEPARATION BY VERTICAL CYLINDER CYCLONE SEPARATOR	
<i>Tri Tjahjono, Marwan Effendy</i>	134 - 143
VARIETY OF HOUSE FUNCTION IN NILASARI HOUSING OF GONILAN, KARTASURA	
<i>Widyastuti Nurjayanti</i>	144 - 151
THERMAL CHARACTERISTICS OF RUSUNAWA - COKRODIRJAN D.I. YOGYAKARTA	
<i>Wied Wiwoho Winaktoe, Angga Ekosaksono</i>	152 - 157
DUCTILITY BEHAVIOR OF REINFORCED CONCRETE COUPLING BEAMS WITH DIAGONAL REINFORCEMENT BETWEEN DEFORM TYPE WITH CRT BAR TYPE	
<i>Yenny Nurchasanah</i>	158 - 167



Prakata

Assalamu 'alaikum wr.wb.

Syukur Alhamdulillah, berkat rahmat dan hidayah Allah S.W.T. Jurnal Teknik Gelagar Volume 17, Nomor 02, Oktober 2006 telah terbit. Hal ini tak lepas dari peran para peneliti dan akademisi yang telah menyumbangkan naskahnya untuk dipublikasikan pada jurnal ini. Pada kesempatan ini Redaksi Jurnal Teknik Gelagar mengucapkan Selamat Hari Raya Idul Fitri 1427 H mohon maaf lahir dan batin, semoga Allah SWT menerima semua amal ibadah kita di bulan suci Ramadhan dan mengampuni semua dosa-dosa yang pernah kita lakukan.

Pada edisi Oktober 2006 ini Gelagar kembali terbit dalam bahasa Inggris. Redaksi telah memilih 10 naskah dari beberapa naskah yang masuk ke meja redaksi. Teknik Elektro menyajikan 3 naskah masing-masing mengenai *Harmonic Power Flow Calculation*, *Electric Propulsion Motors* dan *Voltage Stability Assesment*. Teknik Kimia 2 naskah yaitu mengenai *Peroxide Bleaching* dan *Refinery Gasoline Blending*. Teknik Arsitek 2 naskah yaitu mengenai *House Function* dan *Thermal Characteristics of Rusunawa*. Teknik Mesin, Teknik Industri dan Teknik Sipil masing-masing 1 naskah yaitu mengenai *The Gas Liquid Separation*, *Manufacturing System* dan *Ductility Behaviour of Reinforced Concrete Coupling Beams*.

Sebagai akhir kata, segenap pengurus redaksi mengucapkan terima kasih kepada semua pihak yang telah ikut berperan aktif dalam penerbitan jurnal ini. Redaksi selalu terbuka untuk menerima segala macam kritik dan saran demi kesempurnaan Jurnal Teknik Gelagar.

Wassalamu 'alaikum wr.wb.

Redaksi

BENEFITS OF A CHELATING STAGE PRIOR TO PEROXIDE BLEACHING

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ABSTRACT

The pulping industry has been phasing out chlorine as a bleaching agent and nowadays only a minor part of the production of bleached paper pulp involves the use of chlorine gas. This development has been driven by environmental concerns, because of the formation and emission of chlorinated organic substances that follows the use of chlorine gas in pulp bleaching. The chlorine gas has, to a large extent, been replaced by chlorine dioxide, which generates much lower amounts chlorinated organics than chlorine gas. There are, however, even more environmentally benign bleaching chemicals available, such as hydrogen peroxide. Hydrogen peroxide is nowadays becoming a commonly used bleaching agent in chemical pulp mills. The bleaching efficiency of hydrogen peroxide is, however, dependent on the design of the bleaching sequence in which the peroxide stage is included. In this paper, the importance of a proper chelating stage (treatment with EDTA) prior to peroxide bleaching of eucalyptus kraft pulp is high-lighted. The results show that a high brightness at a high viscosity can be achieved at a relatively low consumption of peroxide, if the bleaching is preceded by a chelation stage. These results are in agreement with results found in the literature. Some results regarding the impact of the alkali addition in the peroxide stage are also included.

Keywords : Kraft pulp, Eucalyptus, *Eucalyptus globulus*, Peroxide Bleaching, Chelating Stage.

INTRODUCTION

The world's paper consumption has increased by 50 % during the last decade. Consequently, the consumption of bleaching agents for pulp bleaching has increased as well. Pulp and paper production has long been recognized as a significant source of pollution. Especially the use of chlorine based bleaching agents has had a negative impact on the

environment. When gaseous Cl_2 is used in bleaching, considerable amounts of organ chlorine compounds are formed (Juuti, S., 1996). Gaseous Cl_2 has largely been replaced by chlorine dioxide (ClO_2) in bleaching in recent years, to reduce the formation of lipophilic organ chlorine and volatile molecular chlorinated compounds. However, the use of ClO_2 in bleaching does not totally eliminate the formation of

some harmful material and from an environmental point of view, it could be advantageous if the use of chlorine dioxide was decreased. (Nakatama, 2004). Society awareness of the environment has urged the use of environmentally friendly compounds in bleaching processes to substitute bleaching sequences including chlorine based chemicals. Such a bleaching concept is usually referred to as totally chlorine free (TCF) bleaching. Hydrogen peroxide (H_2O_2) is one of the key chemicals in TCF bleaching (Van Lierop, 1994). The goals when using this chemical are to maximize delignification, brightness and selectivity towards lignin, and to minimize the consumption of hydrogen peroxide.

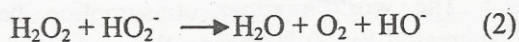
PEROXIDE BLEACHING

Oxygen delignification was industrially established in the 1970's as a method for lowering the kappa number of kraft pulp prior to the bleach plant. Hydrogen peroxide, which is another oxygen based bleaching agent, was introduced at a large scale in bleach plants in the 1990's. There are many similarities between oxygen delignification and hydrogen peroxide bleaching. The pulp is treated under alkaline conditions and molecular oxygen and peroxides are present in both cases, although in different proportions. Hydrogen peroxide is formed in oxygen delignification and oxygen is generated through the decomposition of hydrogen peroxide during peroxide bleaching (Gierer and Imsgard 1977). Compared with oxygen delignification, peroxide delignification appears to provide better color removal, because of the specific action of hydrogen peroxide on chromophores (Dence and Reeve, 1996).

There are, however, some difficulties regarding the stability of the peroxide that have to be considered when hydrogen peroxide is to be used in bleaching. Hydrogen peroxide is very stable under acidic conditions, but under alkaline conditions, it has a propensity for decomposition. The decomposition of hydrogen peroxide is also accelerated by temperature. The active bleaching species in alkaline hydrogen peroxide systems is the perhydroxyl anion, HOO^- , which is formed under alkaline conditions (Dence and Reeve, 1996). The dissociation of hydrogen peroxide (the formation of the perhydroxyl anion) is shown in equation (1).



This anion is a strong nucleophile and is primarily responsible for the bleaching effect of alkaline hydrogen peroxide. There are two competing reactions in hydrogen peroxide bleaching: the first leads to delignification and a brightness increase while a second parallel reaction leads to the decomposition of hydrogen peroxide into water and oxygen. The decomposition is catalyzed by transition metal ions and is usually claimed to involve the formation of reactive intermediates (HO^\cdot , and $O_2^{\cdot-}$). The overall reaction can be written as in equation 2.



Hydrogen peroxide bleaching is usually performed at pH 10-11.5. Under these conditions transition metals can hardly exist as free ions. Collodete et al, (1988) have studied the solubility of Fe^{3+} , Cu^{2+} and Mn^{2+} in the pH range 9.8 to 11.8. After aging at $50^\circ C$ for 120 minutes, the solution

were subjected to ultra filtration (cut off >1000 MW) and an analysis of the filtrates showed that that no amounts of iron, copper or manganese above the detection limit (10 ppb) could be found in the solution after filtration. The rate of peroxide decomposition was shown to be considerably lower in the filtrates than in the original solutions, which implies that the decomposition is surface-catalyzed by colloidal transition metal-oxides/hydroxides. In bleaching, the decomposition is undesirable for two reasons: consumption of peroxide in non-bleaching reactions and formation of hydroxyl radicals ($\text{HO}\cdot$), which oxidize and degrade cellulose. In a study of hydroxyl radical formation by hydrogen peroxide under bleaching conditions, the free hydroxyl radicals are formed by processes catalyzed by mononuclear transition metal ion complexes. Colloidal particles of transition metal oxides/hydroxides were proposed to decompose hydrogen peroxide directly into oxygen and water.

METAL IMPURITIES

The main source of metal ion contamination in pulp bleaching is the pulp itself. Trees (and plants in general) assimilate metal ions and nutrients present in the soil in which they are grown (Young, Guinn, 1966). The amounts and types of metal ions present depend on the species and growing location. Trees grown on the mineral rich soil in Ontario and Quebec (the Canadian shield formation) have a different metal ion content than do trees grown in the Pacific Northwest. Coniferous trees (e.g., hemlock) tend to have a higher metal ion content than do hardwoods such as aspen. The metal ion content of four different wood samples that

accelerate hydrogen peroxide decomposition are listed in table 1. Besides their effect on bleach response and their detrimental effect on hydrogen peroxide stability, metal ions in pulp can form colored complexes with lignin macromolecules. Also of concern is metal contamination from the chemical used to make bleach liquor. Iron is a contaminant in technical grade sodium hydroxide or in bulk sodium silicate. Other non wood sources of metal contamination in pulp include process water and processing equipment.

Table 1. Typical metal ion content of some wood species (Dence and Reeve, 1996).

Wood Species	Metal ion content, ppm		
	Fe	Mn	Cu
Hemlock, western	115	105	14
Hemlock, eastern	20	110	1
Aspen, midwest	55	4	5
Spruce, eastern	17	136	1

The transition metal ions, decreasing the bleaching efficiency in peroxide bleaching, that are routinely encountered are manganese, iron, and copper. Of these, manganese is the metal ion that has received most attention. Other transition metals (e.g. cobalt) also cause peroxide decomposition, but they are usually present in very low amounts in the pulp.

CHELATING STAGE

Some transition metal ions accelerate hydrogen peroxide decomposition (e.g., Mn^{+2} , Cu^{+2} , Fe^{+2}). Concentrations as low as 10^{-9} mol/liter have been shown to effect peroxide stability. Whereas other ions (Mg^{+2} , SiO_3^{2-}) inhibit this acceleration. Ions that accelerate and ions that inhibit hydrogen peroxide decomposition are both found in wood and pulp; unfortunately, the

net end result is a major increase in hydrogen peroxide decomposition (Lapierre, 1995). Therefore, optimal hydrogen peroxide bleaching requires the careful management of the profile of pulp metal ions. The first step in successful hydrogen peroxide bleaching is to minimize the occurrence of catalytic decomposition. This can be achieved by a chelating stage prior to bleaching (Basta et al. 1991).

Cations are surrounded by anions or neutral molecules. The groups immediately surrounding a cation are called ligands, and can be considered as electron pair donors. A ligand molecule may contain two or more atoms which can simultaneously form a two electron donor bond to the same cation. Such ligand molecules are called polydentate ligands or chelates (from the Greek word "chele" meaning claw), as they appear to grasp the cation between the donor atoms. Bidentate, tridentate, quadridentate etc. are terms used to designate chelating agents (chelants) with two, three, four..., binding sites.

Bonding between the electron-donating chelating agent and the electron-accepting metal ion may range from essentially ionic to essentially covalent. Electron donor groups include oxygen and nitrogen and, to a lesser degree, sulphur (Martell and Calvin, 1952). Of all the chelating agents known, ethylenediaminetetraacetate (EDTA) with six binding site is the most frequently used due to its ability to chelate most metal ions in 1 to 1 ratio.

EXPERIMENTAL

The raw material used in this research was oxygen-delignified eucalyptus kraft pulp (*Eucalyptus globulus*) with kappa number: 10.8, brightness: 56.6 % ISO and viscosity: 1195 dm³/kg. This research was

divided in two sections, chelating section and bleaching section.

Chelating stage:

Portions of never-dried pulp (corresponding to 20 g dry pulp) were subjected to different treatments prior to bleaching. After mixing, the treatments were carried out at 10 % consistency in plastic bags placed in a pre-heated water bath for 1 hour at 70°C. Two different kinds of treatments were performed and the performance of the treated pulps was compared with that of an untreated reference.

1. *Reference* No treatment (Sample code:1)
2. *pH 6.1* An end pH of 6.1 was obtained by addition of H₂SO₄ (10 ml of a 5 g/l solution) to the pulp prior to the treatment. (Sample code:2)
3. *EDTA pH 5.5* EDTA (2 kg/ton pulp) was added to the pulp together with H₂SO₄ (10 ml of a 5 g/l solution) prior to the treatment. (Sample code:3-5)

The codes of samples are presented in table 2.

Table 2. The code of sampling stage

Code	Water, g	H ₂ SO ₄ 5g/l,ml	EDTA 5g/l,ml	Wet pulp,g
1	REFERENCE			
2	139.3	10	-----	100.7
3	129.3	10	10	100.7
4	129.3	10	10	100.7
5	129.3	10	10	100.7

After the treatment, the pulps were dewatered and washed carefully. The pH of the filtrate obtained in the initial dewatering was measured.

Bleaching stage

After the treatments described in the previous section, the pulps were subjected to hydrogen peroxide bleaching. The bleaching was performed for 4h and 8h at 10 % consistency in plastic bags placed in a water bath pre-heated to 90°C. Prior to bleaching, the pulp was mixed with H₂O₂ (25 kg/ton) and NaOH (12.5, 15 or 17.5 kg/ton). After the bleaching, the pulps were dewatered and washed carefully. The filtrate obtained from the initial dewatering was used for determination of pH and residual H₂O₂. The pulps were subjected to analyses of brightness, kappa number and intrinsic viscosity.

Determination of residual H₂O₂

Deionised water (30 ml) is put into a Erlenmeyer flask together with 10 ml H₂SO₄ (20 %), 10 ml of sample, 10 ml of KI solution (50 g/l) and some drops of saturated ammonium molybdate solution (catalyst). If hydrogen peroxide is present in the sample, a formation of iodine will take place. The amount of iodine in the solution was determined by titration with 0.1 molar Na₂S₂O₃. The concentration of H₂O₂ in the sample can then be calculated:

$$\text{H}_2\text{O}_2 \text{ g/l} = \left[\text{S}_2\text{O}_3^{2-} \right] \cdot \frac{b \cdot 34}{a \cdot 2} \quad (3)$$

Where, b = volume of Na₂S₂O₃ (ml)

a = volume of sample (ml)

$$\left[\text{S}_2\text{O}_3^{2-} \right] = 0.1 \text{ M}$$

Measuring of brightness

Paper hand sheets were prepared by adding . 5 g of pulp to 1 liter of deionised water, then mixing it by using blender. The pH of the slurry was adjusted to pH 4.5 by adding H₂SO₄ or NaOH. The hand sheet was formed on a filter paper (11 cm diameter) placed in a Büchner funnel

subjected to vacuum. The formed paper sheet was put between two new filter papers (12.5 cm diameter) and then subjected to pressing for 1 minute. When the paper sheet had been dried, the brightness was measured at 457 nm using an Elrepho 2000 instrument.

Kappa number determination

Pulp (between 0.5 g to 3 g, depending on the predicted kappa number) was put into a glass beaker together with 230 ml deionised water. This mixture was mixed with a blender to disintegrate the pulp, then 25 ml of KMnO₄ (0.1 N) and 25 ml of H₂SO₄ (2 M) was added. The reaction was carried out at 25 °C. After 10 minutes, the reaction was terminated by the addition of 6 ml of 1 M KI. The iodine formed was and then titrated with 0.1 M of Na₂S₂O₃. The Kappa number can then be calculated:

$$\text{Kappa} = \frac{(x-y) \cdot d}{m} \quad (4)$$

Where, x = ml of 0.1 N Na₂S₂O₃ that

needed to titrate the blank,

y = ml of 0.1 N Na₂S₂O₃ that

needed to titrate sample,

m = weigh of dry pulp (gram)

d = correction factor.

$$d = 10^{0.00093[(x-y)/0.3-50]} \quad (5)$$

Viscosity of pulp

Pulp (130 mg to 180 mg, depending on the predicted value of viscosity) is put in a plastic bottle (50 ml volume). 25 ml of deionised water and two bars of copper are added, then shake the bottle to disintegrate the pulp. After that, add 25 ml 1 M cupriethylenediamine (CED) solution. Remove air from the bottle, and then shake it in order to dissolve the pulp sample. The intrinsic viscosity is then determined at

25°C according to the SCAN CM 15:88 method.

RESULT AND DISCUSSION

The impact of a chelation stage on the efficiency of the bleaching process is illustrated by the results in table 3,

Table3. The result of bleaching at 90 °C, 4 hours

Code	Kappa No.	Brightness, %ISO	Residual H ₂ O ₂ , g/l	Viscosity, dm ³ /kg
1	9.1	71.5	0	792
2	8.9	72.1	0	731
3	7.8	86.6	1.55	1100
4	7.5	87.3	1.46	1065
5	7.5	87.4	1.36	1049

The addition of NaOH for No. 1,2 and 3 was 15 kg/ton, while for No. 3 it was 12.5 kg/ton and No. 5 it was 17.5 kg/ton.

From table 3 it can be seen that the increase in brightness obtained after bleaching without an EDTA-containing chelating stage is very small, just 15-16 % ISO (No. 1 as reference and No. 2, addition of H₂SO₄, but without EDTA). This moderate brightness gain was reached, even though all of the added H₂O₂ was consumed. It can, however, be assumed that a large part of the peroxide in these bleachings was consumed in decomposition reactions catalyzed by transition metal ions according to equation 2. Furthermore, the decrease in viscosity was very significant for these pulps. There is a correlation between viscosity of pulp and the strength of fiber. A higher value of pulp viscosity implies a lower degree of cellulose degradation and thus a higher fiber strength. Conversely, a combination of H₂SO₄ and EDTA in the chelating stage did substantially increase the brightening effect of the bleaching stage. It can also be

noted that the consumption of peroxide was relatively low when the bleaching was preceded by a EDTA-containing stage. Moreover, the decrease in pulp viscosity was moderate during bleaching of these samples. The consumption of hydrogen peroxide in sample 3 was 44.3 % (of added peroxide) and the increase in brightness was 30.0 % ISO. The peroxide consumption for samples 4 and 5 was 47.4 % and 51.04 %, respectively. The brightness gain obtained for these samples were 30.7 % ISO and 30.8 % ISO, respectively.

Although the highest brightness was obtained in the case of sample 5 (17.5 kg NaOH/ton), the effectiveness of the consumed peroxide may not be optimal, since the largest consumption of peroxide was obtained at this charge of alkali. In order to get more information, additional bleaching experiments, in which the bleaching time was increased to 8 h, were performed. The results are presented in table 4.

Table 4. The results of bleaching at 90 °C, 8 hours

Code	Kappa No.	Brightness, %ISO	Residual H ₂ O ₂ , g/l	Viscosity, dm ³ /kg
3	7.19	88.5	1.21	1033
4	7.14	89.1	1.22	1019
5	7.11	88.0	1.14	1025

From table 4, it can be seen that consumption of hydrogen peroxide in sample 5 is highest, but the brightness gain is lowest. The reason for this is probably that the decomposition of hydrogen peroxide to oxygen and water is promoted by the higher alkalinity.

There is a correlation between kappa number, lignin content and brightness. A high kappa number indicates a high lignin

content. A high lignin content (and a high kappa number) usually indicates a low brightness. The correlation between kappa number and brightness can be seen in figure 1.

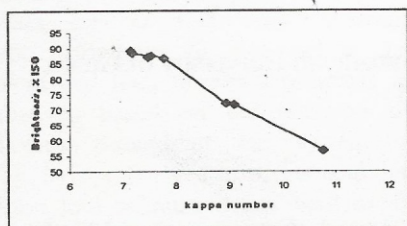


Figure 1. Correlation between kappa number and brightness

A simple, and the most common method, for estimating the lignin content is the kappa number measurement. It is based on the principle that the lignin will consume potassium permanganate at a much higher rate than the carbohydrate components of the pulp; hence, potassium permanganate consumption under carefully specified conditions can be regarded as measure of lignin content in pulp. It should, however,

be considered that also certain carbohydrate related structures in the pulp can be oxidized by permanganate, i.e., hexenuronic acids (Jiebing, Li., 1999).

CONCLUSION

The key to good results in powerful peroxide bleaching stages is to establish an appropriate metal ion profile in the pulp prior to bleaching. This can be achieved by the introduction of a chelating stage before the bleaching. This chelation stage enhances the effectiveness of the peroxide by decreasing the decomposition of peroxide. Not only the consumption of hydrogen peroxide to achieve a high brightness is reduced, but also the loss of viscosity is substantially decreased. Three variables that influence the decomposition of hydrogen peroxide are: metal ion content, temperature and pH. In order to get the best result in peroxide bleaching, an optimization of temperature and pH in the bleaching process is important.

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