A
DISSERTATION REPORT
ON

STUDIES ON PRACTICAL ASPECT OF NEUTRAL AND ALKALINE SIZING

*Submitted in partial fulfillment of the requirements
for the award of the degree of*

**MASTER OF TECHNOLOGY
IN
PULP AND PAPER TECHNOLOGY**

Submitted by
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CANDIDATE'S DECLARATION

I hereby declare that the work, which is being presented in this dissertation report entitled "**STUDIES ON PRACTICAL ASPECT OF NEUTRAL AND ALKALINE SIZING**" submitted in partial fulfillment of the requirements for the award of the degree of Master of Technology in Pulp and Paper Technology, IIT Roorkee, is an authentic record of my own work carried out, under the supervision of Mr. K.RAJ GOEL and Dr. J.S. UPADHYAYA & Dr. NISHI KANT BHARDWAJ, DPT, IIT Roorkee, Saharanpur Campus, Saharanpur.

The matter embodied in the Dissertation report has not been submitted by me for the award of any other degree.

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CONTENTS

1. INTRODUCTION:

Internal sizing is achieved by retarding the rate of penetration of a fluid; usually water, through capillaries formed both within and between fibers. The rate of capillary rise of fluids into paper has been effectively described by the Washburn equation and modifications of it. These approaches assume that the penetration of a fluid into paper is analogous to that of a fluid into a single capillary. Retardation is thus brought about by the creation of a low energy hydrophobic surface at the fiber-water interface which increases the contact angle formed between a drop of liquid and the surface and thus decreases the wettability.

1.1 Why sizing is needed

Papermaking fibers have a strong natural tendency to interact with water. This property is important to the development of strong interfiber hydrogen bond during papermaking and is also the reason that paper losses it strength when saturated with water. This behavior is an advantage for certain absorbent paper grades such as toweling and tissue. Corrugating medium must also be highly absorbent to convert properly in the corrugating process. Such properties are not common for most paper grades, however. It is more typical for papers to interact only moderately with water during use. Table 1 lists some common examples.

Table 1 Paper end uses and converting requirements needing liquid penetration resistance

- Off machine coating
- Offset printing
- Liquid packaging
- Gluability
- Wet / frozen foods packaging
- Water based ink holdout

Liquid penetration resistance is important to many papermaking processes where paper is brought into contact with aqueous solutions, such as size pressing, coating, and calender water box applications. Finally, many papers (e.g. office papers, cereal boxes, cosmetic boxes, grocery bags) must be able to resist accidental wetting during use.

1.2 Types of sizing processes

There are two types of sizing processes.

1>Internal sizing: -when chemicals are added to the papermaking furnish and retained on the fibers by appropriate means in the wet end of the system, the process is termed "internal sizing" the purpose is to modify the surface of the fibers to control penetration of the aqueous liquids into the paper.

2>Surface sizing: -In this process, the paper web is formed and dried and a sizing agent is applied to the paper surface by a size press, coater, or calender box.

Table 2 lists some of common materials used for these two purposes. This discussion will focus on internal sizing.

Table 2 some typical materials used for internal and surface sizing

Internal addition	**Surface treatment**	
To reduce Water wettability	**To reduce water wattability**	**To reduce pore radius**
Emulsion size	Reactive size	Carboxyl methyl cellulose
Reactive size	wax emulsion	Styrene-Maleic Anhydride
		Copolymers starches
Rosin size		
Wax emulsion		
Stearates		
Fluorocarbons		

2. Basic surface science considerations important to sizing

We are interested in the rate and extent of liquid (usually water) penetration in to paper under variety of conditions. Such phenomena come under the general surface and colloid science heading of capillarity.

Four ways that liquid penetrates paper: it has been hypothesized that liquid moves through paper by the following three mechanisms:

- Capillary action through pores
- Diffusion through pores
- Diffusion through fiber solid fraction

It was also suggested that a fourth mechanism involving evaporation, vapor phase diffusion, and condensation could occur.

2.1>Capillary flow in pores: the Washburn equation

Since a paper web can be considered to consist of a series of interconnected pores, the most attention has been paid to capillary flow in pores. More than fifty years ago an equation, which has become known as the Washburn Equation, was developed to describe the penetration of liquid into fiber beds.

In its differential form, the Washburn equation is the following:

$$dl/dt = \gamma r \cos\theta / 4\eta l$$

Where l =the capillary distance penetrated in time, t; and r is the capillary radius; η is the liquid viscosity; γ is the liquid surface tension; and θ is the contact angle between the capillary walls and the liquid.

It is assumed that no significant external pres sure exist on the penetrating liquid and that the penetrated structure does not change with time.

2.2 Contact angle and surface wetting

The term "wetting," repellency," and "penetration" are all relative once. There will always be some, at least slight attraction between a liquid and a solid in contact. No surface actually exercises a repulsive force upon a liquid. The practical distinction between a surface wetted by water and another not wetted is that the former allows water to spread over it as a continuous film, while the water on a non-wetted surface rest in small, separate droplets. At the air – water – solid interface, these droplets form a contact angle.

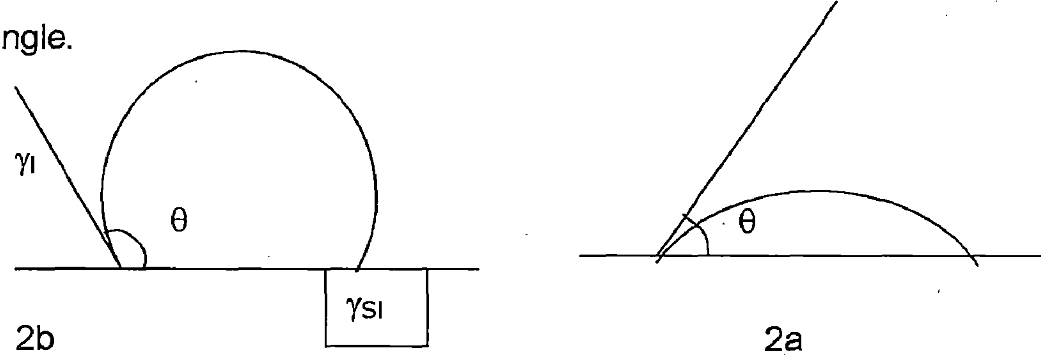


Figure-1

On an ideal (clean, smooth, homogeneous, insoluble) plane surface, force balance around the droplets, which can form a acute contact angle as in fig 2a or an obtuse angle as in fig 2b. When the contact angle is less than 90° the liquid is capable of the wetting of solid surface, while at larger angles wetting does not occur. When a finite contact angle θ is formed by a drop of liquid, the relationship between the surface tension of a solid, liquid, and the interfacial tension between the solid and the liquid is known as **Young's equation,** usually expressed in an approximated form:

$$Cos\theta = (\gamma_s - \gamma_{sl})/ \gamma_l$$

Where γ is interfacial surface tension, and s, l and sl are subscripts referring to the solid and liquid, and the solid – liquid interfaces.

3. Requirement of good internal sizing agent

We have defined sizing is a process whereby a sizing agent is applied to papermaking fiber to lower their attraction for liquid water. Another way to look at this say that sizing makes a paper surface more hydrophobic by reducing its surface tension (or surface energy). It is generally accepted that to be good sizing agent, a material must meet the following criteria.

Table 3. Requirement of a good internal sizing agent

In order to be a good internal sizing agent, a substance must:

- Have high retention on fiber
- Be uniformly distributed on fiber surfaces
- Be able to produce hydrophobic fiber surfaces
- Adhere strongly to fibers
- Be chemically inert to the penetrants of interest

In addition to above five criteria, it is also desirable that a sizing agent has no adverse effects on the papermaking process.

3.1 High retention of size on fibers

It is obvious that unless a sizing agent is retained in the paper making web during sheet formation it can not be effective. The primary retention mechanism employed in internal sizing is the beterocoagulation of size particles with fibers and fibers fines.

In order to promote this phenomenon, it is necessary to impart a positive charge to size particles so that they will attracted to, and retained by, negatively charged fibers and fines. As will be discussed below. Common sizing agents are either anionic or nonanionic in natural form. Consequently, It is necessary to use a cationic retention aid to promote size retention. Many different materials can server this purpose, such as alum, cationic synthetic polymer, and cationic starch. In all instances, they must react with the neutral and anionic size particles to produce positively charged particles entities that will be attracted to fibers and retained by them.

3.2 Uniform distribution of size on fiber surface

For the most part, uniform distribution is promoted by multiple addition locations at the addition point, good addition point mixing, low size concentration, and low consistency. The charge difference between fibers and size needs to be balanced so that size particles retention rate is not too high or the mixing forces will not have time to be effective.

3.3 Ability to produce hydrophobic fiber surfaces

This property is an absolute requirement of sizing agents because, as was described above, sizing agents work by increasing the hydrophobicity of paper. Most common sizing molecules have a dual polar:nonpolar nature and are called **amphipathic** molecules. The nonpolar part usually composed of C's and H's --- provides the required hydrophobic character.

In addition to having hydrophobic character, it is necessary that mechanism exist by which the sizing molecules will orient is hydrophobic part away from the fiber surface. In this manner, liquid molecules impinging on the paper surface will "see" the hydrophobic size molecules instead of the water-attracting fiber surface cellulose and hemicellulose molecules.

3.4 Ability to adhere strongly to the fiber surface

A fourth requirement is that the oriented size molecules must become anchored to the fiber surface so that they do not overturn when contracted by water.

3.5 Be chemically inert to the penetrant

This is a logical requirement. If the sizing agent reacts with the penetrants, then it could be converted to a nonhydrophobic form or be released from the fiber surface and lose its effectiveness. Fortunately, water is the principal penetrants of interest, and it is a relatively benign substance.

3.6 Adverse effects on the papermaking process

This requirement is listed separately in table 4 because, in practice, it cannot be met all of the time for any sizing agent. All common sizing agents have attributes that can lead to papermaking process problems under certain conditions. The goal is to minimize these problems through the informed use of sizing materials.

4 Internal sizing

The purpose of internal sizing is to modify the surface of paper to control water penetration, in order to either limit the pickup in a converting operation, e.g., at the size press or coater, or to impart water repellency to the end product. The three most common internal sizing agents currently being used and treated in this text are rosin systems, alkyl ketene dimmers(AKD), and alkenyl succinic anhydrides(ASA).

way to obtain resistance to aqueous penetration, although conversions from acid to neutral or alkaline pH in papermaking in many cases have necessitated the use of specisl sizes such as alkyl ketene domer or alkenyl succinic anhydride.

5 Sizing at neutral or alkaline pH

5.1 Reasons for neutral-alkaline papermaking

The 1980's saw the break through of neutral-alkaline papermaking. Although several factors have influenced the decisions, the primary considerations in changing from acid to neutral-alkaline papermaking relates to the possibility of processing furnishes with a content of calcium carbonate. Calcium carbonate is today the filler of choice for fine paper grades. Moreover, the use of calcium carbonate pigment as a high opacity pre coat as the principal coating pigment is growing steadily, regardless of the coated paper or broad grade or type of process used.

Table-4. Some potential advantages and drawbacks of neutral papermaking.

Process benefit	**Product benefit**
Calcium carbonate can be used as filler and in coating	Improved strength properties. The increased strength potential alternatively permits higher content of filler e.g.,hardwood
Reduced energy consumption in refining and drying	Higher filler content affords better opacity
Higher drainage rate, easier drainage	Good printability,e.g.,bulk,brightness
Less buildup of inorganic solubles	Calendering without blackening possible at higher moisture levels
Specific water consumption can be Reduced	Improved paper performance
Less corrosion	Improved chemical resistance, e.g., against lactic acid, alkalies
Process drawbacks	**Product drawbacks**
Synthetic internal sizes required. Hydrolysis of can cause problems.	Size curing can be incomplete, difficult adjusting the degree of sizing.
Temperature limitations in the wet-End	Size reversion and fugitivity can embarrassing customer complaints.
Acid (pH <4) chemical pulps in integrated mills can create problem.	Fine filler required for opacity

High load of anionic trash with mechanical pulps	Yellowing with mechanical pulp if pH >7.5
Deposit and picking problems; wear and plugging of wires and felts High microbiological activity, slime	Slippery sheet surface at hard sizing
Cumbersome optimation of retention Systems.	Large dosage of OBA can interfere marketing efforts and strategy.
Chemical incompatibility, e.g., Restricted use of Alum, more expensive dyes and wet-strength resins required, OBAs are ineffective	(Low paper gloss)

Calcium carbonate in any system, if allowed to equilibrate, produces an alkaline pH. Recycling of coated broke therefore tends to present problems in an otherwise acid process owing to the decomposition of the CaCO3 presents. The decomposition already begins gradually at pH6-6.5, resulting in accumulation of sparingly soluble gypsum and calcium hardness in the white water, and liberation of carbon dioxide. Alum, for example, will give cause to the following reactions:

$$3CaCO_3 + Al_2(SO_4)_3 + 3H_2O \Leftrightarrow 2Al(OH)_3 + 3CaSO_4 + 3CO_2 \text{ --------------(5)}$$

$$CaCO_3 + CO_2 + H_2O \Rightarrow Ca(HCO_3)_2 \Leftrightarrow Ca^{2+} + HCO_3^- \text{ -----------------------(6)}$$

$$CaSO_4 \Leftrightarrow Ca^{2+} + SO_4^{2-} \text{ ------------------------- (7)}$$

Solubility 2gms/l

The stiochiometric consumption according to reaction (4) of technical grade aluminum sulfate is about 2kg per kg of calcium carbonate. Side reactions(5) and(6) are also unacceptable, with saturation by gypsum and very high calcium hardness in the aqueous phase. Thus large quantities of calcium carbonate in broke and white water can cause severe pH swings, foam, poor retention, and pinholes in contact with alum.In the field of wood-free fine paper, the neutral-alkaline papermaking technology using either natural ground calcium carbonate(GCC) or synthetic precipitated carbonate(PCC) as filler and coating pigment, seems to be relatively well mastered.Consequently the internal sizing of alkaline fine paper is today effected exclusively by application of synthetic agents such as alkyl ketene dimmer(AKD) or alkenyl succinic anhydride(ASA). AKD use is more common in europe, while in the united state the application of ASA is growing and today surpasses that of AKD.

5.2 Neutral rosin sizing

Not until 1980-1985, when the mechanism of rosin sizing became accepted, could rosin systems be logically designed to also function under neutral conditions. For neutral pH conditions, the key conception is that the agent used, alum and size, do not necessarily have to react in the wet end-that the reaction between adsorbed hydroxylaluminium floc and resin acid also in a neutral process takes place in dryer section of the paper machine under the influence of heat. The logical requirements are as follows:

1. To restrict any extensive depression of pH in order to preclude the reactions with calcium carbonate. If possible, this should be accomplished by minimizing the dosage of the Al-source chemical.
2. To create aluminum species which are sufficiently cationic to be adsorbed by anionic surfaces in the pH region 6.5-7.5.
3. To use or develop a modified dispersed resin acid to minimize ionization in the neutral pH region.
4. To use a retention aid capable of retaining the anionic resin acid close to the adsorbed cationic aluminum floc.

To maintain a sufficient Al-level without decreasing the pH too much, condition1 has been met in practice also by substituting part of aluminum with sodium aluminate. In any case, a trasient pHchanges will occour from neutral to slightly acid and back to neutral. The process is therefore called pseudo-neutral.

As far as condition 2 is concerned, when alum is added to a furnish at pH 6.5-7.5, the Al-species will be ultimately converted to aluminum hydroxide which, however, does not lose its charge all at once

Condition 3 has been met successfully by the producers of rosin dispersion by development of special although more expensive formulations.

Condition 4 is not a "problem" but has meant more careful attention on application parameters such as coagulation retention systems, filler type and usage, and chemical addition sequences.

5.3 Alkaline sizing

While many synthetic organic molecules are capable of imparting water resistance to paper, only ASA and AKD have been widely adopted by the paper industry and there is approximately equal usage of the two materials today in US alkaline fine paper manufacture. In Europe and other paper making regions, the relative use rates of AKD and ASA differ from 50:50, but they still remain the preeminent alkaline sizing materials.

5.4 Alkaline sizing requirements

In order to be a good sizing agent, a substance must:

- Have a high retention on fibers
- Be uniformly distributed on fiber surfaces
- Be able to produce hydrophobic fiber surfaces
- Adhere strongly to fibers
- Be chemically inert to the penetrants of interest

Requirement one – Since ASA and AKD are nonionic molecules that have no innate attraction for anionic fibers, other substances, such as cationic starch and synthetic cationic polymers, must be used as size retention aids.

Requirement two - ASA and AKD occur as a water insoluble solids or liquids at room temperature – depending upon the length and degree of unsaturation of their hydrocarbon chains.In order to good distribution of these substances on the fiber surfaces.

Requirement three – ASA and AKD molecules contain relatively inert hydrocarbon chains that impart hydrophobic character to the materials and enable them to do the same for fiber surfaces.

Requirement four – The molecules contain reactive centers that react with fiber surface hydroxyl groups to form covalent linkages that are stable in the presence of water and anchor the size molecules to fibers.

Requirement five – ASA and AKD are the capable of providing sizing against more aggressive liquids than rosin sizes. This is a result of their strong bonding to fiber surfaces and the ability of their hydrocarbon chains to affect large areas of the fiber surfaces.

6 Sizing with alkenyl succinic anhydride

6.1 Emulsification

The alkenyl succinic anhydride (ASA) is composed of an unsaturated alkenic hydrocarbon backbone coupled to succinic anhydride. The products are usually manufactured in two stages: First an unsaturated linear or branched olefin, example-an 1-alkene is isomerized by moving its double bond randomly from its α-position. This will yield an ASA product that is liquid at room temperature, a condition important for easy emulsification of the size on application at the paper mill. In the second step, the mixture of isomerized alkenes is reacted with maleic anhydride to produce the ASA raw material proper.

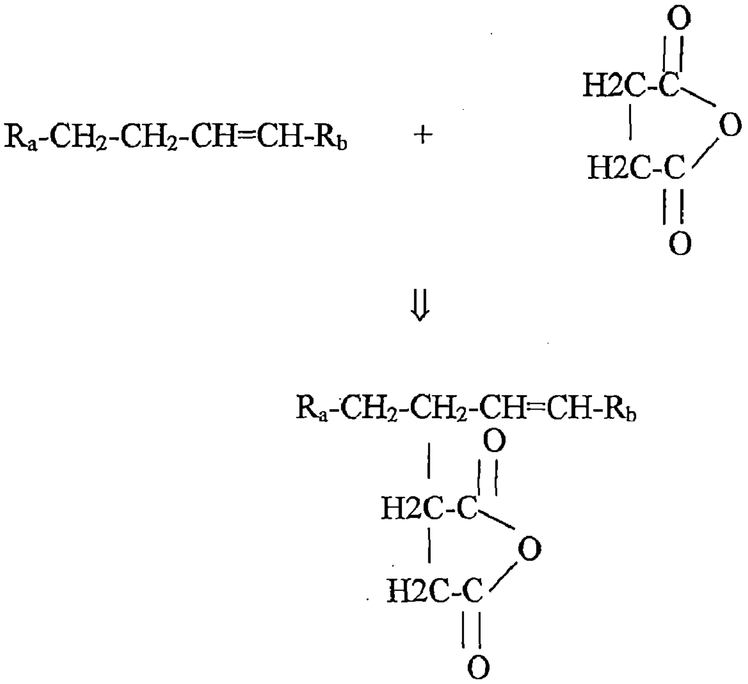


Figure-2

Normally ASA is a yellowish oily product. This 100% active substance can be store as such for long time but must be well protected from water or humidity. It will not dissolve in water and, prior to application in papermaking, must be emulsified onsite at the paper mill. Activators are surface-active agents that promote effective emulsification under condition of low mechanical energy. Emulsion stability is best with synthetic polymer , and sizing efficiency is best with cationic starch improving with the amount of starch used; consequently, propriety mixed emulsification systems have been introduced. The ratio of starch to ASA is usually in the range 2:1 to 4:1.

The emulsification process imparts a cationic charge on the oil droplets which helps to increase the stability as well as improves ASA retention on anionic fibers and sizing efficiency. The synthetic cationic polymers used enhance these properties and are often called "promoters". The emulsification process must be adjusted accordingly whenever a different ASA is used.

6.2 Chemistry and application of ASA

ASA undergoes the normal reaction of acid anhydrides. Of particular interest in conjuction with sizing is the reaction with alcholic hydrixylgroups to yield an ester, and the hydrolysis with water. Both reactions occur in papermaking system.

ASA is highly reactive, and the reactions proceed rapidly and irreversibly. Although this would provide satisfactory development of sizing on a paper machine, the hydrolysis of ASA is undesirable. If a mediocre ASA is used, more ASA will be needed to achieve the sizing desired and more ASA will be lost by hydrolysis and, consequently, increases the probability of problems.

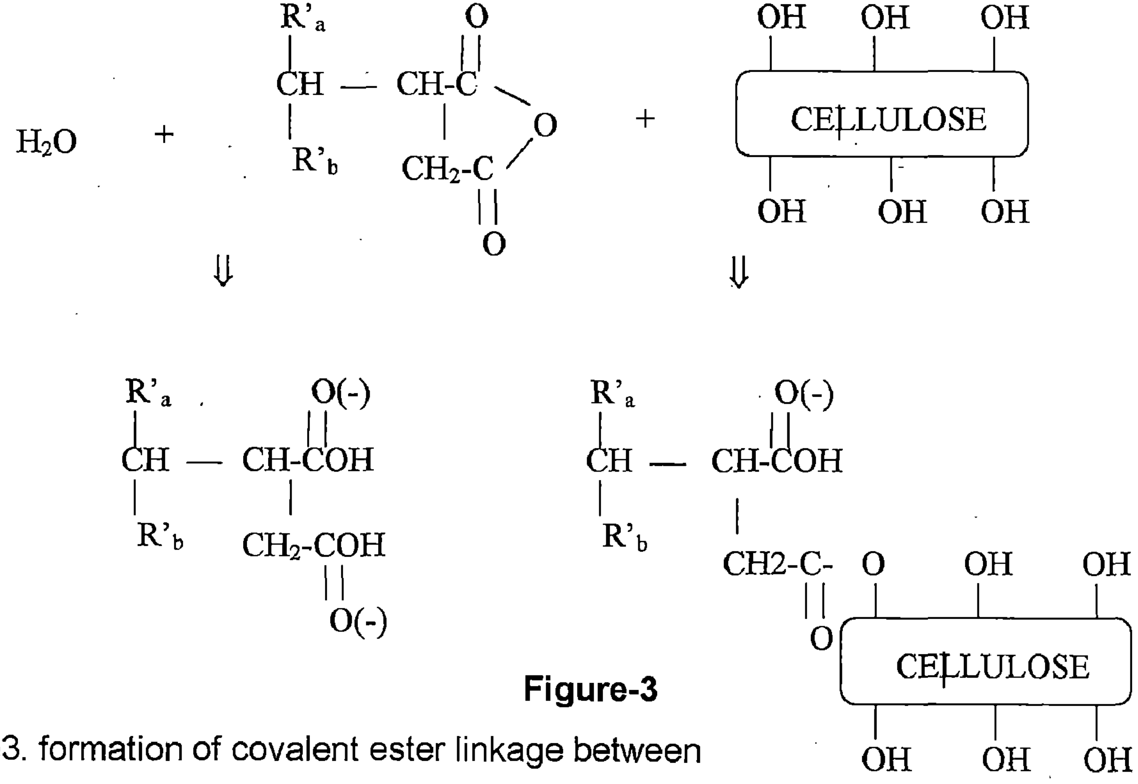


Figure-3

Figure3. formation of covalent ester linkage between

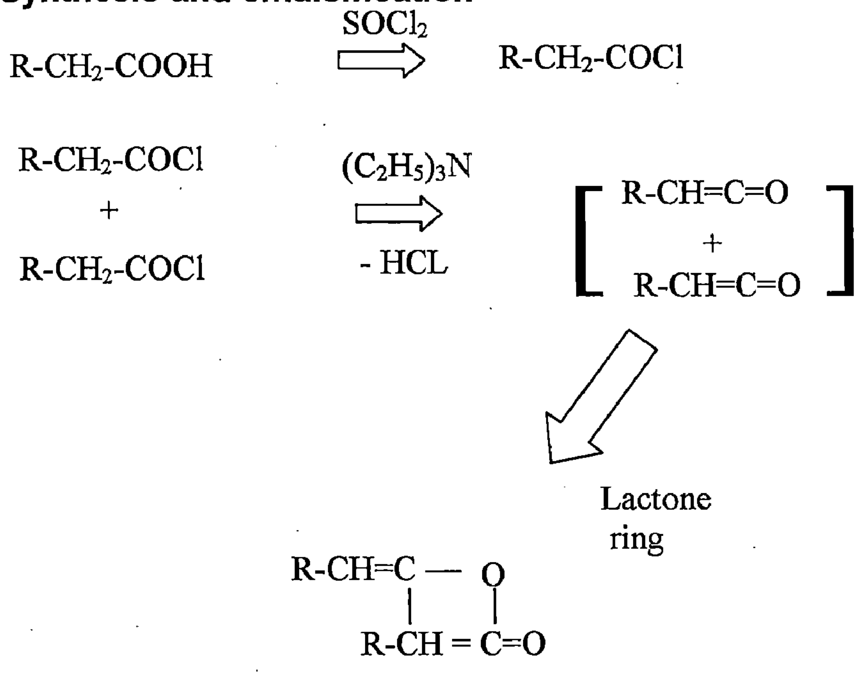
A cellulose hydroxyl group and anhydride portion of ASA(right), and competing hydrolysis of ASA into a dicarboxylic acid (left).

The hydrolysis of ASA accelerates with pH, time, and temperature. In order to limit the hydrolysis of ASA emulsion, pH can be lowered immediately after the emulsification by addition of aluminum sulfate. The time lag between prepration and use should be as short as possible.

The hydrolysis decomposition of ASA still continues after the dosage into the furnish and can even accelerate because usually the pH and temperature of the stock are higher then in the imulsion proper. Addition points should be selected to minimize the residence time. A long residence time will cause size hydrolysis and lead to deposit problem. Thus the machine chest is not recommended as an addition point. Addition of the emulsion here is said to provide the highest sizing with the least sizing variation. Other suitable locations for ASA dosage, with good mixing for satisfactory distribution of emulsion particles, are the inlets to the fan pump and screens.

7 Sizing with Alkyl ketene dimmer

7.1 Synthesis and emulsification



ALKYL KETENE DIMER

Figure-4

Structurally, alkyl ketene dimmers (AKD) are unsaturated lactones. A frequently used synthesis route involves the preparation of the acid chloride of a carboxylic acid (accomplished with, e.g., phosphorus trichloride, phosphorus pentachloride or thionyl chloride), followed by intermolecular lactone ring condensation via a labile intermediate carboxylic acid obtained by dehydrohelogenation of the acid chloride with trianolamine in an organic solvent.

The emulsification is usually effected in a hot (75°C-90°C) solution containing the cationic starch stabilizer and a small amount of surfactant, e.g., sodium lignin sulfonate, to which are fed AKD flakes. After melting of AKD, the mixture is forced through microfluidizers and cooled. Small amount of a promoter (low MW cationic polymer with high charge density) and biocide can also be included. the particle size of the dispersion produced is today normally 0.5 to 2 microns, with a mean weight average near 1 micron. Although a low melting point aids in emulsification, it can be a drawback later in hot paper making systems where the temperature can rise above the melting point, and the stability of the dispersed particles can be destroyed on account of the melting of AKD inside.

Recent advances in AKD synthesis and emulsification technology have made it possible to produce dispersion at considerable higher solid content than earlier, the dispersions are now in the market with even 20%-25% solids. Usually about 20%-40% of the solids content is cationic starch and/or some other more strongly cationic polymeric stabilizer. The charge of the AKD dispersion can accordingly vary from slightly to highly cationic.

The dispersion delivered to customer have pH ranging 2.5 to 3.5, owing to acidification with either sulfuric or hydrochloric acid. This prolongs shelf life by reducing potential hydrolysis of the AKD. At pH > 6, it becomes more reactive by opening of the lactone ring in the molecule. Because of the acidity, corrosion resistant equipment is needed for storage and pumping. The AKD producers usually store and ship the AKD below 20°C.

7.2 AKD reactivity

The AKDs are believed to react with the hydroxyl group of cellulose to form β-keto esters (fig-5). However, AKD also react with water under the formation of an unstable β-keto acid which decarboxylates to the corresponding ketone, e.g., stearone or palmitone (fig-11)

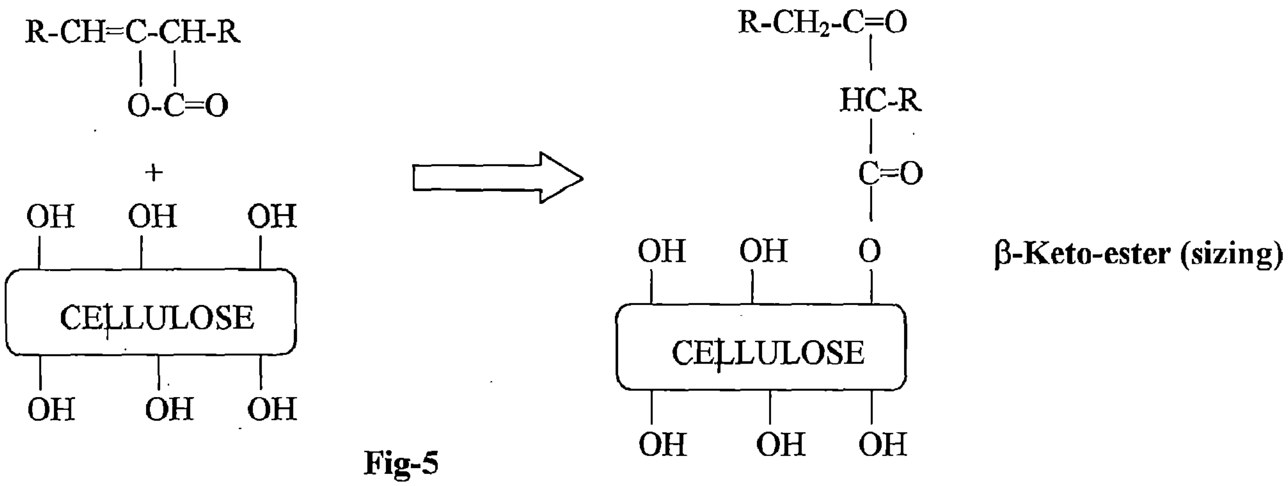


Fig-5

Reaction between AKD and the hydroxyl groups of cellulose with formation of a β-keto ester linkage.

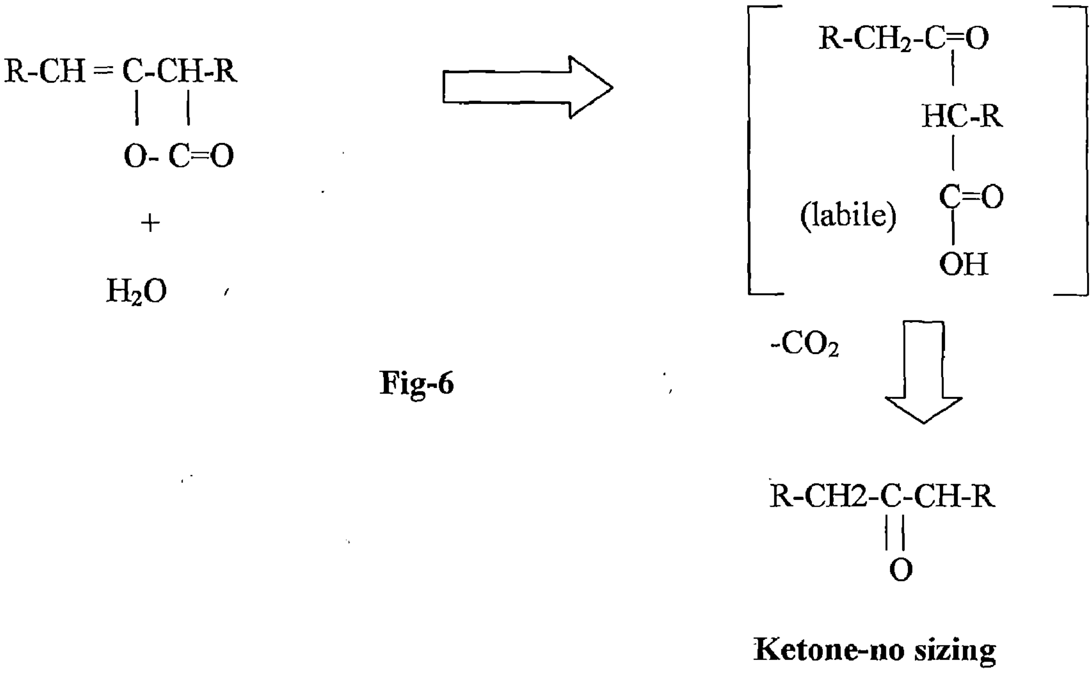


Fig-6

Figure-6 Hydrolytic decomposition of AKD via a labile β-keto fatty acid into ketone (e.g., stearone, palmitone).

In comparison with ASA size, AKD is considerable less reactive. The competing hydrolysis reaction was critical with earlier AKD-dispersions. However, the stability of most commercial products has been improved significantly in recent years as far as hydrolysis is concerned. Contrary to the hydrolysis product of an ASA size that is always lost in the white water, the ketone resulting from the hydrolytic decomposition of AKD is a solid (melting point 75°C - 80°C), which can be retained in the sheet but does not contribute to sizing. Thus the risk of deposit formation is less with AKD, although the labile intermediate β-keto acid, if formed in the aqueous phase, can form relatively stable tacky precipitates with calcium ions.

Opinions differ in regard to the extent of reaction of AKD with cellulose. Although conclusive evidence of a β-keto ester link is hard to obtain, the covalent bond mechanism has been reasonably well accepted. Data and findings that support the occurrence of chemical reaction between AKD and cellulose are more frequently than work implying that β-keto ester formation shown in fig –10 might not occur.

Both views are based upon thorough laboratory investigations, by application of sophisticated methods, e.g., use of radioactively labeled AKD, and GC, DSC, FTIR, ESCA, or NMR techniques. It is an undisputed fact the unreacted AKD adsorb on to the fiber surface also possesses sizing properties. Removal of any unreacted AKD by extraction with organic solvents such as tetra hydrofuran (THF) or chloroform and investigation of whether treated material still possesses sizing properties or retention irreversibly bound AKD thus are integral parts of most of the studies.

7.3 Mechanism of interaction

The interaction between AKD and the cellulose fiber proceeds in four stages (fig-7);

1> The dispersed, cationically stabilized particles are first adsorbed onto the fiber surface by electrostatic attraction. The AKD – dosage point for carrying out this step varies and lies typically within the area machine chest ⟷ fan pump ⟷ level box. Additional cationic starch can be added to assist the retention of AKD either before or before with the dosage of the AKD.

2> As the web is being heated and dried in the paper machine, the adsorbed AKD wax starts to melt and after spreading covers at least part of the fiber surface as a thin layer. This intimate contact with the cellulose fibers will eventually make chemical reaction possible.

3> The chemical reaction with the cellulose hydroxyls, however, can not proceed to any marked extent or at a sufficient rate until the major proportion of the water has been evaporated from the web. As for web moisture content, the start and rate of reaction will depend upon pH, alkalinity, and temperature.

4> As the chemical reaction gradually proceeds under the influence of heat, there occurs an energetic re-arrangement of the molecules so that the hydrophobic ends of the molecules turn out from the surface, thus imparting water repellency to the web. Also this curing can also proceed at a low rate in the paper reel at the pope and storage.

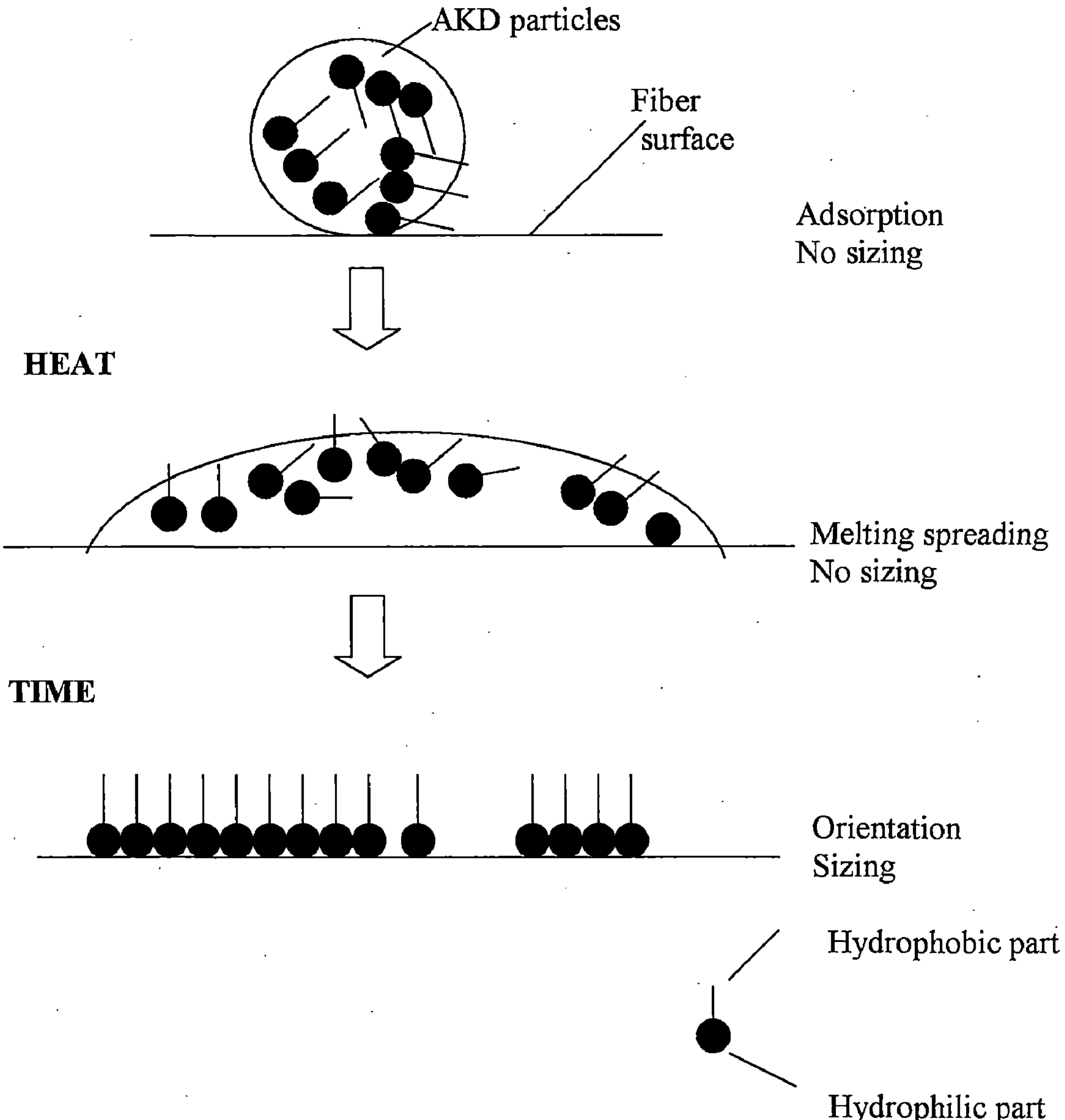


Figure--7

While it appears understandable that AKD must spread on the surface of fibers before any esterification could take place, there is no fundamental reason that this should be a

Prerequisite also for the hydrolytic interaction of AKD with water. The extent and rate of the competing hydrolysis of AKD are also favored by pH and heat but still require the presence of water. According to Marton the rate of hydrolysis increases more steeply than the rate of esterification, and the hydrolysis in mill system becomes a significant factor. The ketone forming side reaction, in addition to losing cellulose –reactive groups in the expensive size, can and does cause undesirable performance problems. Care therefore should be taken to design the web drying profile in a way that expels excess water as early and quickly as practicable.

Also curing must be initiated appropriately because chemical reaction alone is insufficient for good sizing. If on-machine surface sizing or coating will be carried out, curing should preferably be complete prior to the treatment concerned. For these reasons, it is common to dry the web to very low moisture content before the size press, typically to 3%-5% but many times even 1%-3%, especially on fast running machines. This can assist greatly but, even under these conditions, there will still considerable off-machine development of sizing.

7.4 Some factors affecting AKD sizing

Fillers, fines and retention

Fillers and fines increase the amount of AKD required in much the same way as is the case with size demand in rosin sizing and ASA sizing. This is primarily due to the increased surface area to be sized. The differences in sizing response at low and high PCC surface area in the absence and presence of cationic starch. It is clear that AKD is very efficient at sizing pulp in the absence of both starch and PCC. Calcium carbonate filler, and its high surface area modification, in particular, greatly impairs sizing response. When cationic starch is added, it enhances the sizing by preferentially depositing the AKD on the fiber surfaces. It was noted in the same study that the total retention of AKD is a function of starch does while the percentage of that AKD is chemically bound is a function of the charge level of the starch.

pH and alkalinity

Alkalinity is a measure of the buffering capacity of the aqueous system principally due to the concentration of bicarbonate ions (HCO_3^-), expressed as the equivalent amount of calcium carbonate in mg/L (ppm). The effect of pH and alkalinity on AKD sizing are interrelated. The pH of the papermaking system affects sizing but not independent of alkalinity

Alkalinity effectively contributes to the reaction between AKD and cellulose. The underlying mechanism is not fully understood, but the bicarbonate ions have been found to exercise a proton-transfer catalytic effect. A moderate alkalinity level (e.g.,50-200ppm) is generally considered favorable if not indispensable, as curing otherwise can fail.

At satisfactory alkalinity, the effect of pH upon sizeability is quite clear : Although the rate of cure increases with pH, post cure will usually proceed satisfactorily to practically the same end point at any pH higher than 7. Soluble alkalinity is the easiest way to ensure a stable, buffered system pH, to increase the sizing efficiency of AKD, and to counteract negative effects of alum. Table –5 presents effect of pH and alkalinity upon off-machine sizing and post cure.

Table-5. The influence of pH and alkalinity upon the development of sizing.

		Hercules size Test (85% reflectance), s					
			After				
pH	Soluble alkalinity ppm	Off machine	4h	8h	24h	1 week	2 weeks
6	0	1	1	2	28	204	310
7	0	1	11	44	136	409	423
7	50	35	152	267	383	490	517
7	150	69	221	299	381	536	575
8	0	22	99	151	288	429	434
8	50	191	348	445	514	577	601
8	150	366	515	601	572	582	601
9	0	95	233	253	324	415	405
9	50	244	412	462	488	539	524
9	150	389	558	630	556	538	590

High alkalinity (in excess of 400 ppm) should be avoided- it is most often due to a high concentration of hydroxide ions and high pH favoring the hydrolysis of AKD, and can also cause reversion of sizing (= loss of size response with time after the paper has reversion and previously been observed by increasing the alkalinity from 100 to 1100 ppm. This produced a complete loss of sizing response at accelerated oven aging within the first seven days.

Sources yielding high alkalinity are sodium hydroxide, calcium hydroxide, and overdosage of sodium carbonate Na_2CO_3. Insufficient alkalinity is best corrected by self adjust in respect of both pH and alkalinity, and dosage of additional bicarbonate ions is not required.

7.5 Fugitive sizing and size reversion

The term "fusitive sizing" and "size reversion" in relation to poor or normal sizing and size efficiency are often used in a confused way. The term can be defined as follows.

Size efficiency: -Size efficiency is the relationship between the size response (as determined by an appropriate method of measurement) either before an external size press or at the reel or after laboratory oven cure and the amount of size added to the papermaking system.

Normal: -Normal or good sizing is the ability to achieve in-specification paper at the reel and to maintain it over time.

Poor sizing: -Poor sizing is when paper is out of specification at the reel; however, an oven cure might or might not show in- specification paper. Upon natural curing of the reel sample, the paper remains out of specification.

Size reversion: -Size reversion is when paper is in specification at the reel and, upon aging, the sizing level decreases to a level where it then remains constant.

Fugitivity: - Fugitivity or loss of sizing, results when paper is in specification at the reel and, upon natural aging, all of sizing is lost. Hence size reversion and fugitivity are different issues.

7.6 Other potential papermaking problems related to AKD.

Papermakers have also experienced other problems when converting from acid to alkaline papermaking. Some problems directly associated with AKD sizing include:

Wet press picking – This problem often occurs in the early stage of conversion to AKD sizing and alkaline papermaking when rosin containing broke and stock is still in the system. It has no proven to be a major obstacle to conversion in most instances.

Insufficient sizing ahead of the size press – Only moderate sizing is required to achieve good size press runnability and by controlling pH, alkalinity, and drying within required ranges, this should be possible with cationic AKD sizes.

Paper slipperiness – AKD sized paper often has a lower coefficient of friction than rosin sized paper due to the fact that AKD is a waxy material. If high AKD levels are used in order to achieve high sizing levels, than the paper surface can become too slippery for certain converting and handling process. There is also evidence that the AKD ketone hydrolysis product contributes to this problem.

7.7 Comparison of AKD and ASA attributes

Table- 6. Comparison of AKD and ASA

Attribute	AKD	ASA
Reaction rate	Slow to moderate with fibers and water	Very fast with fibers and water
Physical rate	Solid product and hydrolyzate	Liquid product and hydrolyzate
Hydrolyzate reactivity	Unreactive	Cause deposit problems and is a desizing agent
As required	Emulsion	Oil that requires in mills emulsification
Impact on precision converting grades	Critical that addition rate be kept below levels that cause slippery paper	Addition rates not as critical because slip problems not prevalent
Effective pH range	Neutral to alkaline ph levels	pH rang 5-9
Hard sized grades	Able to do without significant problems	Difficult without deposit problems due to high dose levels

7.8 Recyclability of AKD-sized paper

The major proportion of the sizing potential of any AKD that ends up in the white water loop will be lost on account of the hydrolysis into ketone. On the other hand, very little is known about the fate of AKD on recycling of sized paper. It can be surmised that a significant proportion of the unreacted AKD will be lost by hydrolysis because of the relatively long chest hold time in warm water. What about the reacted, covalently bound AKD, Obviously the first study on this interesting topic was published recently by Sjostrom and Odberg.

8.0 STUDIES ON PRACTICAL ASPECT OF NEUTRAL AND ALKALINE SIZING

Bleached mixed hard wood kraft pulp was collected from the stock preparation section of **BALLARPUR INDUSTRIES LTD., YAMUNANAGAR**. The pulp was collected after refining. The freeness of the pulp was 32 °SR and brightness was 89 %ISO.

FURNISH OF RAW MATERIALS

Poplar	20%
Eucalyptus	15%
Bamboo	20%
Veneer waste	45%

The chips of above wood were charged in the digester and cooked at 6.8 kg/cm^2 pressure and 160°C temperature by kraft process with white liquor containing 16% active alkali (Na_2O) and sulphidity was 22% and liquor to wood ratio 1:2.7

P.No of unbleached pulp	12
Bleaching sequence	$CDEO_pD_1D_2$
Yield of pulp (bleached)	43%
Viscosity of the pulp	11cps

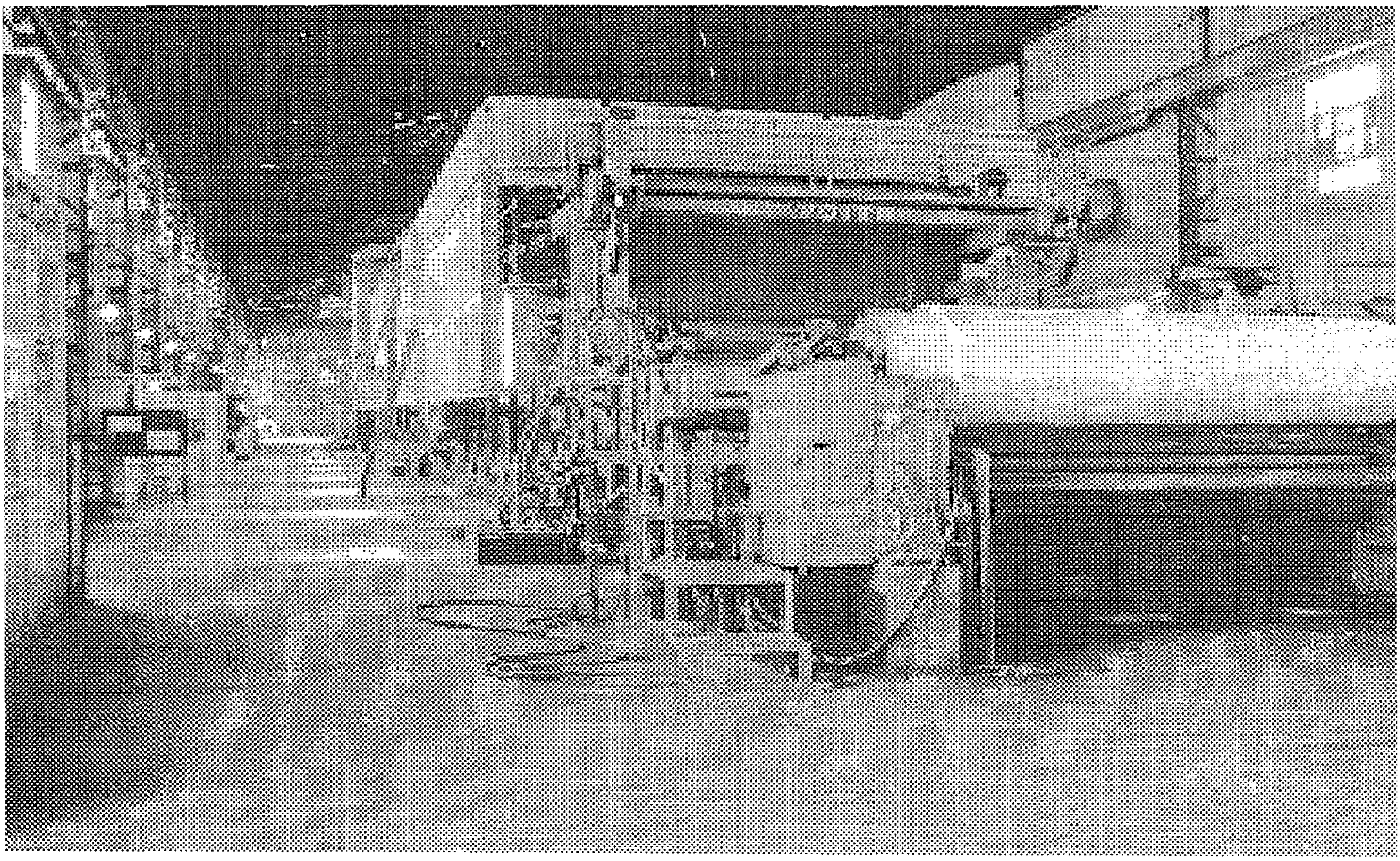
8.1 PLANT TRIAL No. 1 of Alkaline sizing (AKD) at M/c No. 7: We have taken plant trial to replace Neutral sizing (Excel & PAC) by alkaline sizing (AKD).

Objectives

- To change over from neutral sizing to AKD sizing.
- To improve paper Brightness and permanency of paper.
- To reduce sizing cost.

First we specify the machine -7 to take the plant trial

➤ **Machine specifications**

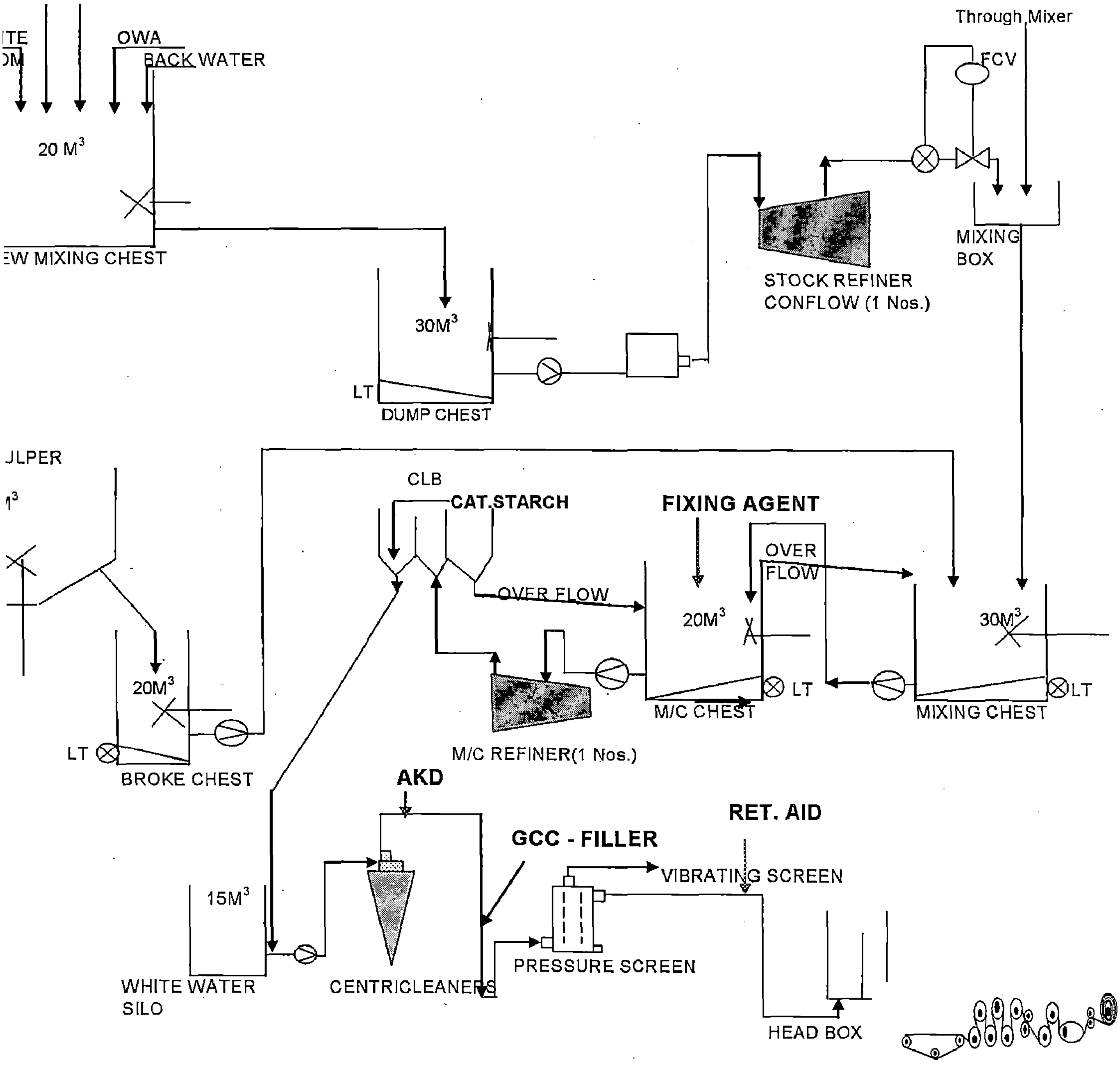


Make : Karulla
Type : MF with Size press & Kuster calendar
Speed : 120 m/min
Grammage : 70-100 g/m^2
Deckle : 2.72 m
Quality : Royal Executive Bond Paper - 70 gsm

AKD sizing with dosing points

AKD : Modified fatty alkyl diketene with medium cationic charge. **(Primary centricleaning accept)**
Retention Aid : Cationic polyacrylamide base. **(Outlet of pressure screen)**
Fixing agent : Polyvinylamine base. **(Machine chest)**

8.2 Wet-end flow diagram: Dosing points



8.3 Chemical dosage and Cobb values

	Neutral size	PAC	AKD-3	Fixing agent	Ret. Aid	Cobb values , g/m²				FPR	FPAR
	kg/MT	kg/MT	kg/MT	g/MT	g/MT	Before size press	As such at pope reel	Cured Oven 105±2	After 24 hrs	%	%
ze	32.5	38.5	-	-	-	30/32	22/24			80.3	52.6
	32.5	38.5	-	-	-	-	19/20			83.7	55.5
	32.5	38.5	-	-	-	27/29	23/25			82.9	49.0
	32.5	38.5	-	-	-	-	24/26			84.0	54.2
	32.5	38.5	-	-	-	29/31	24/26			84.2	50.4
	32.5	38.5	-	-	-	-	25/27			82.8	52.7
D	32.5	38.5	8.0	300	50	27/29	26/28	26/27	-	82.9	48.0
	28.1	32.8	8.0	300	50	30/31	26/27	23/25	-	83.0	51.2
	28.1	32.8	8.0	300	50	-	25/27	22/23	-	83.0	52.0
	14.4	19.8	12.0	350	50	48/50	32/34	19/21	-	81.8	52.9
	14.4	19.8	12.0	350	50	-	41/43	21/23	-	83.7	51.3
	14.4	19.8	12.0	350	50	60/62	43/45	21/23	21/23	84.3	52.9
	0	21	18.0	350	50	-	40/44	20/22	-	84.8	48.4
	0	21	18.0	350	40	85/95	62/66	21/23	22/24	83.3	50.0

size		3	g agent	Aid						R
kg/MT	kg/MT	kg/MT	g/MT	g/MT	Before size press	As such at pope reel	Cured Oven 105 ±2	After 24 hrs	%	%
0	21	16	350	20	-	83/85	21/23	22/24	84.0	51.2
0	21	16	350	20	96/99	80/82	21/23	21/24	83.8	52.4
0	21	16	350	20	-	96/98	24/26	22/24	84.0	52.4
0	20	16	300	20	94/99	98/102	22/24	21/23	81.6	56.9
0	20	18	300	20	109/111	101/102	21/23	22/24	83.6	56.1
0	20	18	300	20	-	109/112	23/25	22/24	84.8	57.0
0	20	20	300	20	120/130	120/129	28/30	25/28	85.0	56.5
0	20	20	300	20	-	118/122	26/29	26/28	84.6	55.8
0	20	18	300	20	98/104	96/99	23/25	22/24	85.2	56.0
0	20	16	300	20	-	86/90	23/25	22/24	84.8	54.6
0	20	16	300	20	80/85	64/67	18/20	20/22	84.5	52.0
0	20	16	300	20	52/56	42/44	19/20	19/21	83.8	52.4
0	20	16	300	20	-	94/97	23/25	21/23	83.3	52.0
0	20	16	300	20	54/56	44/46	22/24	21/23	86.3	56.4

8.4 Backwater pH, Conductivity, Alkalinity, Hardness & Charge demand

ple	Back water pH	Conductivity m S/cm	M Alkalinity as CaCO3	Hardness as Ca	Charge demand meq/l	
					Head box	Back water
	7.1	1140	210	126	18	23
	7.3	1150	215	135	18	22
	7.4	1020	230	152	17	19
	7.4	1090	220	130	17	21
	7.3	1100	232	128	11	22
	7.3	1170	232	170	13	18
	7.4	1040	215	160	10	17
	7.4	1140	230	140	26	30

ductivity : Checked with conductivity indicator controller instrument , linity : T 553 om.00, Hardness :Book quantitative inorganic analysis, gel IV 18 EDTA method , Charge : Particle Charge Analyzer.

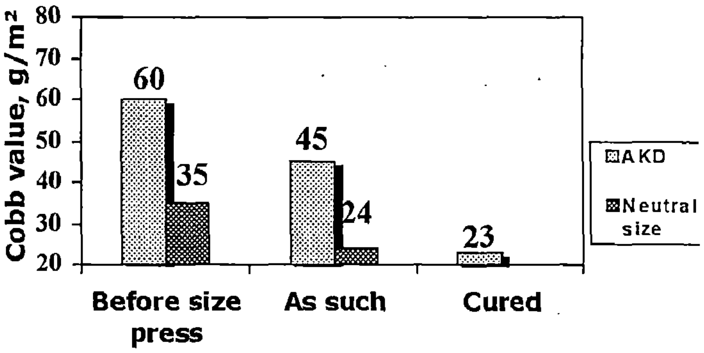
8.5 Surface temperature profile of dryer cylinders.

Dryer no.	Before trial, Temp. °C	AKD trial,Temp. °C			
1	74	70	70	72	
2	84	90	88	83	Before press
3	84	88	91	87	
4	85	90	91	93	
5	84	86	87	84	
6	82	88	87	90	
7	92	92	93	93	
8	94	96	93	98	
9	93	95	97	92	
10	92	102	101	98	
11	75	68	79	75	
12	80	81	78	84	After press
13	92	90	87	84	
14	94	86	88	80	
15	103	96	96	83	
16	93	94	93	79	
Steam pressure kg/cm²	**1.67**	**1.94**	**1.94**	**1.87**	

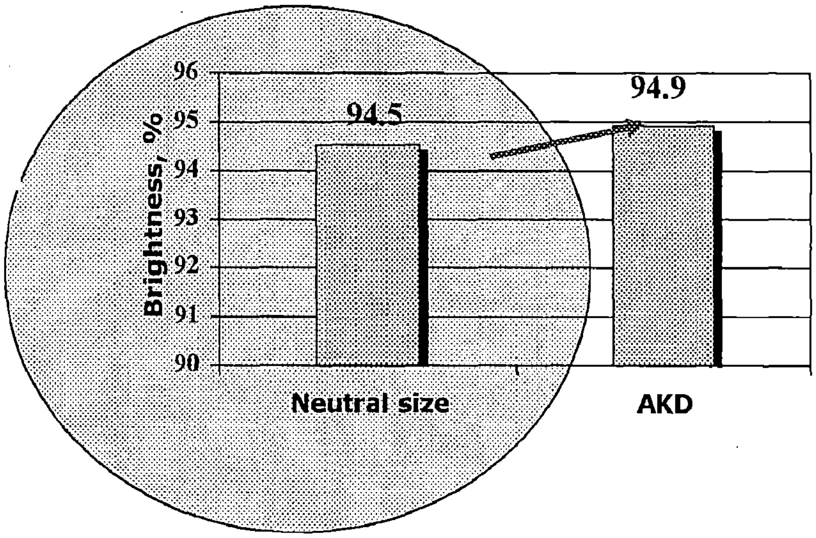
8.6 Paper properties

Parameters	**Neutral size**	**AKD Trial**
Substance, g/m^2	70.1	70.0
Bulk, cc/g	1.42	1.42
Burst Factor	25.6	25.5
Breaking length,m, MD/CD	6100/3200	6150/3210
Cobb , as such, g/m^2, T/W	22/24	40-80/45-100
Cured Cobb, g/m^2, T/W	-	19-23/23-25
Smoothness, ml/min. top/wire	100-250 200-320	100-250 180-320
Stiffness, taber, MD/CD	2.0/1.0	2.1/1.0
Ash, %	11.6	11.5
Brightness,ISO	94.5	94.9
Opacity, ISO	85.0	85.1
CIE-Whiteness	150	150
Formation index	150-160	150-160
PC No.	0.50	0.35

Cobb values with Neutral size and AKD run



Effect on ISO Brightness



8.7 Observations of plant trial: -

- Hardness, alkalinity and conductivity of backwater were in the desired range.
- Back water pH was in the range of 7.1 to 7.4 .
- Surface temp. of dryer cylinders before size press was in the range of 70 to 102° C, which affected the curing rate of AKD.
- Steam pressure variations were observed in the range of 1.67 to 2.0 kg/cm².
- Head box charge was in the range of –110 to –190 mv and corresponding cationic demand was in the range of 10 to 26 meq/l.
- Paper brightness increased 0.4 point and all other paper properties were comparable.

- Wide variation in Cobb values at pope reel i.e 40-100 g/m². However, cured Cobb values remained in the range of 19-25g/m².
- The Cobb values before size press were in the range of 60-110 g/m².
- Higher AKD dosage up to 20 kg/MT did not result in lower Cobb value.
- Whenever, Cobb increased up to 90 g/m², there was problem of paper feeding through size press roll at the time of paper break.
- Machine runnability was poor and some times breaks were observed after size press roll in the third dryer group due to high Cobb before size press. Paper used to become slack in the 3rd dryer group

8.8 CONCLUSIONS: -

- Lower dryer temperature, due to low steam pressure, resulted in higher Cobb values before size press.
- The desired Cobb value before size press should be around 50-60 g/m² for better machine runnability.
- The desired Cobb values of 20-25 g/m² after curing could be achieved even at high as such Cobb value at pope reel.
- PM-7 has drying limitation as regards to AKD sizing of paper.

8.9 Recommendation / Future plan

- Due to drying limitation, the optimization of AKD could not be done .
- AKD consumption was more and finally there was marginal reduction in sizing cost i.e. Rs. 55.0 / MT.
- Further trial will be conducted after steam pressure is increased to a level of 2.5 - 3.0 kg/cm² for faster curing and saving in AKD so that cost effectiveness is obtained.

9.0 Trial No. 2 of Alkaline sizing (ASA+AKD) of M/s IVAX at PM-4:

1. Objectives

   - To change over from neutral sizing to Alkaline sizing
   - To reduce sizing cost per ton of paper.
   - To use GCC/PCC in place of soapstone.

2. Background

To derive the benefits of alkaline sizing during paper making, trial was conducted at PM-4 in Apr.08 with M/s IVAX. At present, we are using dispersed rosin as sizing chemicals in pH range of 6.5-6.7.

3. Trial Plan

A) Chemical details and dosing points

- IVAX 111 (ASA) : Alkenyl Succinic Anhydride.
- IVAX 999(Emulsifier) : Cationic polymer.
- IVAX 750(AKD) : Alkyl Ketene Dimer Emulsion .
- IVAX Poly TC (Fixing agent) : Cationic liquid polymer.
- IVAX TIN 100(Flocculant) : Cationic Polyacrylamide
- IVAX print jet (Surface size) : Styrene Acrylate Copolymer.

Chemicals	**Concentration gpl**	**Dosing point**	**Responsibility**
IVAX 111 (ASA)	10	Pressure screen inlet/ Sec. Fan pump Inlet	By IVAX
IVAX 999 (Emulsifier starch)	500	ASA Emulsifier	By IVAX
IVAX Poly TC	50	Mixing chest	By IVAX
IVAX 750 (AKD)	As such	Mixing chest	By IVAX
IVAX Print Jet	As such	Size press service tank	By IVAX
PAC	Regular	M/C Chest	By BILT
IVAX RTN 100	1.0	Pressure screen In let	By IVAX
Press roll cleaning	As suggested by supplier	Lump breaker roll, Ist & IInd press top roll	BY BILT
Felt cleaning	As suggested by supplier	All press felts	BY BILT

B) Parameters monitored

The following parameters were measured.

- Pulp pH
- Back water pH
- FPR and FPAR
- SR of refined pulp and head box
- Chemical consumption
- Machine conditions-couch vacuum, dryer temperature and steam pressure.
- Physical and optical paper properties.

9.1 Blank (without AKD+ASA) Neutral Sizing

Annexure-1

Date	28.3			29.3			30.3			31.3			1.04		
Time	A	B	C	A	B	C	A	B	C	A	B	C	A	B	C
M/c speed, mpm	340	340	340	340	340	335	337	337	340	340	340	340	340	340	340
GSM	64	64	64	68	68	80	54	54	54	54	60	60	60	60	60
Quality	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC	BC
Finished Draw, MT/hr	3.6	3.6	3.6	3.6	3.6	4	3	3	3	3	3.5	3.5	3.5	3.5	3.5
PULP															
PH	6.55	6.7	6.5	6.55	6.7	6.6	6.52	6.6	6.8	6.85	6.7	6.7	6.55	6.7	6.7
Brightness	89.2	84.6	88	90.2	90	85	89	90	89	89	89	90	89.2	90	90
O SR after refining	28														
O SR M/c chest	34														
O SR H/B	44														
H/B pH	6.4			6.3			6.5			6.4			6.3		6.3
B/W pH	6.6			6.6			6.8			6.6			6.6		6.6
B/W Alkalinity	60			65			58			52					
FPR	76.0			69.6			72.9			75.7			77.1		76.8
FPAR	44.6			43.3			45.0			37.0			40.4		42
Broke															
Flow addition per batch m3	15	15	15	15	15	15	15	15	15	15	15	15	15	15	10
Alcofix kg/MT	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10	0.10
Chemicals															
PAC LPM	1.3	1.3	1.3	1.6	1.6	1.6	1.2	1.2	1.4	1.2	1.2	1.2	1.2	1.2	1.2
PAC kg/MT	29.9	29.9	29.9	36.8	36.8	33.1	33.1	33.1	38.6	33.1	28.4	28.4	28.4	28.4	28.4
Filler flow LPM	65	65	64	65	66	65	70	30	25	25	26	25	25	27	25
Filler kg/MT	217	217	213	217	220	195	280	120	100	100	89	86	86	93	86
M/C parameters															
Couch vacuum	460	400	450	480	450	460	400	500	400	400	450	440	400	400	450
Dryness after couch	19												19.1		20
Dryness after press	38												40.7		39

Dryness before size press	93												94		94
Dryness final	96												96.4		97
Steam flow, T/hr	13	12	12	12.8	13	13	13	11	11	10.8	12	11	11.2	11.4	12
Properties															
Ash%	16.2	17.6	16	16.6	17	12	13	11	12	11	11	10	14	14	14
Cobb, before size press TOP	28														
WIRE	36														
As such cobb TOP	19	18	19	21	20	19	21	18	28	19	21	22	20	19	22
WIRE	22	22	22	29	24	22	24	22	37	22	27	26	23	25	25

C) Execution

Before staring the trial, we collected data during normal dispersed rosin sizing. At present rosin size consumption was 10.0 kg/T, PAC 29.0 kg/T at back water pH of 6.5-6.7. The detailed data are given in Table under **Blank (without AKD+ASA)** above annexure-1.

During ASA+AKD trial run, we proposed to maintain Cobb of approx. 60 g/m^2 before size press to avoid breaks at size press. The cured Cobb of 25 g/m^2 was targeted at pope reel.

Trial of ASA+AKD started on 3.04.08 at PM-4 in 60gsm Bilt Classic paper after the wash up (5 hrs) of machines. During trial, the wire passivation and lump breaker passivation chemicals of M/s Amazon chemicals were started, initially both ASA and AKD were added. The ratio of ASA and Starch was 1: 2.

9.2 ASA+AKD with retention aid

In the beginning of trial, the dosage of chemicals were ASA at 0.5kg/T, AKD at 8 kg/T, Retention aid 17g/T, Poly TC fixing aid 220 g/T and Print jet 1.1 kg/T of paper produced at machine. Dosage of retention aid was very low i.e. 17 g/T only. After 3-4 hrs, most of the chemicals were reduced i.e. ASA 0.5 kg/T, AKD 4.0 kg/T, Retention aid 10-15 g/T, Poly TC fixing aid 200 g/T and Print jet 1.1 kg/T. Back water pH was in the range of 6.5-7.0. , PAC consumption was 10-15 kg/T. Head box charge was -65 to -85 mv with 11-25 meq/l charge demand. Average FPR and FPAR was 80% and 46%, 76% and 43% and 78% and 48% in 70gsm, 78gsm and 80gsm respectively. Cobb before size press varied from 51 to

78g/m^2 and cured Cobb of 15-17g/m^2 i.e. paper was hard sized (as such Cobb at pope reel was 20-30 g/m^2). This combination was run for about 20hrs in Bilt Classic 70, 78 and 80gsm paper and approx. 80MT paper was manufactured. Breaks were observed due to picking at lump breaker and 1st press. However, all paper properties were normal. Looking into the picking problem, the retention aid and fixing agent were stopped to reduce FPR and FPAR.

Avg. wet-end data and avg. GSM wise paper properties are given in Annexure - 2 & 6 respectively.

Wet-end and machine parameters during the ASA+AKD trial and ASA in BILT CLASSIC at PM-4

with retention aid

annexure-2

Date	3-4.04.08	4.04.08	4.04.08
M/c speed, mpm	335.0	335.0	335.0
GSM	70.0	78.0	80.0
Quality	BC	BC	BC
Machine Draw, MT/hr	4.5	5.0	5.1
PULP			
pH	6.6	6.5	6.4
Brightness	87.4	88.5	88.5
o SR after refining	31.0	32.0	23.0
o SR M/c chest	35.0	35.0	31.0
o SR H/B	46.0	42.0	42.0
H/B pH	6.7	6.3	6.5
B/W pH	7.0	6.5	6.8
B/W Alkalinity, ppm	46.0	32.0	42.0
H/B charge, mv	-95.5	-89.0	-75.0
Chage demand, meq/l	28.5	20.0	18.0
FPR	79.8	75.9	77.9
FPAR	46.3	43.1	47.7
Broke			
Flow addition per batch m3	11.9	10.0	13.0
Alcofix kg/MT	0.1	0.1	0.1
Chemicals			
ASA LPM	3.7	4.0	3.8
ASA kg/MT	0.5	0.5	0.5
AKD IVAX 750 LPM	0.4	0.4	0.4
AKD IVAX 750 kg/MT	5.5	4.2	4.3
IVAX RTN 100 lpm	2.3	2.5	2.5
IVAX RTN gm/MT	15.3	15.0	14.6

IVAX poly TC lpm	0.3	0.3	0.3
IVAX poly TC g/MT	215.0	180.0	194.0
IVAX Print Jet LPM	0.2	0.2	0.2
IVAX Print Jet kg/MT	1.1	1.0	0.9
PAC LPM	0.7	0.7	0.7
PAC kg/MT	12.6	11.7	11.4
Filler flow LPM	65.2	71.0	71.0
Filler kg/MT	174.1	170.1	165.8
M/C parameters			
Couch vacuum	418.0	450.0	400.0
Dryness after couch		19.2	
Dryness after press	40.5	39.4	
Dryness before size press	95.6	92.8	
Dryness final	96.6	96.8	
Steam flow, T/hr	14.1	14.8	13.9
Properties			
Ash%	15.8	18.0	16.5
Cobb, before size press TOP	60.7	66.5	64.1
WIRE	63.9	76.5	67.3
As such cobb TOP	19.5	20.3	20.4
WIRE	22.9	24.0	23.9
Cured cobb TOP	14.6	16.9	17.2
WIRE	15.7	18.4	18.2
cobb after 24hrs TOP	15.2	18.0	16.5
WIRE	16.4	19.0	17.8

9.3 ASA+AKD without retention aid

To eliminate press picking and reduce retention, the retention aid and fixing agent were stopped. ASA and Starch ratio was reduced from 1:2 to 1:1.5. During this period, chemical addition was as follows. ASA 0.3-0.4 kg/T, AKD 2-4 kg/T and Print jet 1.3 -2.0 kg/T. Back water pH was in the range of 6.5-7.1. PAC consumption was 20-40 kg/T. Head box charge was -20 to -62 mv with 9-12 meq/l charge demand. Average FPR and FPAR was 77% and 39%, 78% and 43%, 78.9% and 41.7%, 81% and 44.5% in 60gsm, 64gsm, 68gsm and 80gsm respectively. Cobb before size press varied from 62 to 98g/m^2 with cured Cobb of 16-25g/m^2 i.e. some time hard sized paper was manufactured (as such Cobb at pope reel was18-60 g/m^2). This combination was run from 6.0PM of 5.04.08 to 3.0PM 8.04.08(69 hrs). All grammage of Bilt Classic were manufactured. About

200 MT of paper was manufactured. The major observations during this period were:

- Machine runnability was normal. However, some times picking at press was observed.
- At times Cobb of approx. $90 g/m^2$ was observed before size press that resulted in breaks at size press.
- Cured Cobb was mostly in the range of 16-18 g/m^2.

Wet-end data and grammage wise paper properties are given in Annexure -2*& 6 respectively.

Wet-end and machine parameters during the ASA+AKD trial and ASA in BILT CLASSIC at PM-4

Annexure-2*

without retention aid

Date	7-8.04.08	5,6,7,11.04.08	6,7,11,12.04.08	6-7.04.08	5-6.04.08
M/c speed, mpm	335.0	335.0	335.0	335.0	335.0
GSM	54.0	60.0	64.0	68.0	80.0
Quality	BC	BC	BC	BC	BC
Machine Draw, MT/hr	3.5	3.9	4.1	4.4	5.1
PULP					
pH	6.5	6.6	6.5	6.7	6.7
Brightness	87.8	88.1	88.0	88.6	87.9
o SR after refining	38.0	38.0	40.0	30.0	24.0
o SR M/c chest	37.0	34.0	37.0	36.0	33.0
o SR H/B	51.0	48.0	48.0	48.0	43.0
H/B pH	6.2	6.5	6.6	6.6	22.9
B/W pH	6.5	6.8	6.8	6.8	6.8
B/W Alkalinity, ppm	52.7	60.0	89.0	52.0	15.1
H/B charge, mv	-55.7		-27.0		-56.0
Chage demand, meq/l	13.3				13.5
FPR	80.6	77.1	77.7	78.9	81.0
FPAR	40.7	39.0	42.9	41.7	44.5
Broke					
Flow addition per batch m3	9.5	10.0	9.6	10.0	8.9
Alcofix kg/MT	0.1	0.1	0.1	0.1	0.1
Chemicals					
ASA LPM	1.8	2.5	2.5	2.5	2.5
ASA kg/MT	0.3	0.4	0.4	0.3	0.3
AKD IVAX 750 LPM	0.2	0.3	0.3	0.3	0.4

AKD IVAX 750 kg/MT	5.5	3.8	3.3	3.8	4.7
IVAX RTN 100 lpm					
IVAX RTN gm/MT					
IVAX poly TC lpm					
IVAX poly TC g/MT					
IVAX Print Jet LPM	0.3	0.3	0.3	0.3	0.4
IVAX Print Jet kg/MT	1.7	1.7	1.6	1.6	1.6
PAC LPM	1.0	1.3	1.4	1.3	0.9
PAC kg/MT	22.8	27.0	28.8	25.2	15.2
Filler flow LPM	10.1	32.1	37.4	40.7	59.3
Filler kg/MT	34.9	100.1	109.1	111.8	140.9
M/C parameters					
Couch vacuum	444.8	470.0	452.0		
Dryness after couch	19.1				
Dryness after press					
Dryness before size press	95.4				
Dryness final	96.4	96.4	96.5	96.7	
Steam flow, T/hr	11.5	11.2	12.0	12.6	15.5
Properties					
Ash%	10.9	12.3	13.3	14.0	13.8
Cobb, before size press TOP	71.3	73.7	63.3		66.5
WIRE	76.6	77.6	68.7		80.0
As such cobb TOP	44.7	26.7	50.4	38.4	59.1
WIRE	49.9	31.8	54.8	43.5	64.1
Cured cobb TOP	19.1	16.2	18.0	17.2	19.2
WIRE	21.8	17.0	20.8	18.5	22.7
cobb after 24hrs TOP	22.8	15.3	19.5	18.5	18.4
WIRE	24.9	16.4	20.7	20.1	20.5

Paper properties during the ASA+AKD trial at PM-4:Bilt Classic

Annexure-6

Properties		Unit	ASA+AKD						
DATE			7,8,11,12,19,21.	5,6,7,.04.08	22.4.08	5,6.04.08	3,4.04.08	4.04.08	5,6.04.08
Lot/roll			4848,57,70,73	4843,45	4875	4841,46,69	4838	4840	4839,42
Substance	NOM	g/m²	54	60	64	68	70	78	80
	ACT		54.5	61	65.5	68.6	71.4	80	80.8
Thickness		Micron	78.9	86.8	90.5	95.4	98.4	107	113.7
Bulk		cc/g	1.44	1.42	1.38	1.39	1.38	1.34	1.41

Bust factor			29	26.5	24.7	27.1	25.5	28	23.1
Tear factor	MD		49.5	47.4	46.7	50.8	45.4	49.7	51.1
	CD		53.7	52.1	54	55.3	49.7	52.3	53
Breaking length	MD	Meter	7343	6619	7525	6987	6326	6424	5991
	CD		3970	3740	3947	3438	3776	3173	2882
Double Fold	MD	no.	20	13	17	17	23	22	12
	CD		12	8	10	9	12	10	7
As such cobb	Top	Gm/m²	47.4	19.4	51	19.5	22.8	16.5	27.5
	Wire		52.4	22.4	55	24.6	24.6	18.5	33.8
Cured Cobb	Top	Gm/m²	19.6	15.8	18.8	16.1	15	14.5	18.2
	Wire		22.1	17	22	17.6	16.2	17.8	19.4
Wax pick		No.	14	14	14	13.3	14	14	13
Ash		%	8.1	14.6	14.2	13.9	15.7	19.5	15.5
Gurley porosity		Sec/100 ml	15.9	14.2	17.2	17.2	14.6	24.8	16.3
Brightness	ISO	%	88.7	89.6	90.4	89.9	89.7	89.6	90.5
Opacity		%	85.8	87.9	90.3	90.1	90.8	93.1	93
Yellowness		%	-13.6	-13.5	-13.5	-13.7	-13.7	-13.7	-13.7
Fluorescence		%	19.2	18.5	18.7	18.5	18.7	18.8	18.4
CIE whiteness		%	145.4	145.2	145.6	146.1	146.1	146.2	146.6
Smoothness	Top	ml/min	60-140	60-160	50-160	60-160	60-160	70-140	60-160
	Wire		100-200	100-280	80-240	120-240	130-260	80-200	130-260

It was then decided to run 100% ASA.

9.4 100% ASA

To overcome the problem of picking at pH of 6.9-7.0, the trial of 100% ASA was taken. ASA was added at the dosage of 0.40 to 0.70 kg/T. During this period, print jet at size press was kept 2.0 to 3.0 kg/T. Back water pH was in the range of 6.5-7.0. PAC consumption was 20-40 kg/T. Head box charge was -60 mv with 15 meq/l charge demand. Average FPR and FPAR was 81% and 42%, 82% and 41% , 81% and 47%, 77% and 44.5%, 76% and 45%, 79.5% and 53% in 54gsm, 60gsm, 64gsm, 68gsm, 70gsm and 80gsm respectively. Retention was high as compared to ASA+AKD. Cobb before size press varied from 69 to 77 g/m² with cured Cobb of 18-30g/m² (as such Cobb at pope reel was 20-60 g/m²). This combination was run for 2½ days (i.e. 8-10.04.08). All grammage of Bilt Classic were manufactured. Total 177 MT paper was manufactured. The major observations during this period were:

- For about one day, the machine runnability was normal. As the ASA was hydrolyzed in the Backwater and broke pulp, severe press picking was observed.
- In this case also, as the back water pH increased (7.0), picking problem increased at the lump breaker and 1st press and it also resulted in high cured Cobb i.e. the range of 30-35g/m².
- Chemical coating at size press roll was observed.

Wet-end data and grammage wise paper properties are given in Annexure no. 2** & 7 respectively.

Wet-end and machine parameters during the trial 100% ASA in BILT CLASSIC at PM-4

Without retention aid Annexure-2**

Date	8-9.04.08	10-11.04.08	9,11.04.08	10.04.08	9.04.08	9.04.08
M/c speed, mpm	335.0	335.0	335.0	335.0	335.0	335.0
GSM	54.0	60.0	64.0	68.0	70.0	80.0
Quality	BC	BC	BC	BC	BC	BC
Machine Draw, MT/hr	3.5	3.9	4.1	4.4	4.4	5.1
PULP						
pH	6.6	6.6	6.6	6.3	6.4	6.5
Brightness	88.7		88.5			
O SR after refining	36.0	37.0	33.0	33.0	29.0	32.0
O SR M/c chest	39.0	36.0		32.0	33.0	
O SR H/B	37.0	50.0	46.0	48.0	46.0	48.0
H/B pH	6.4	6.3	6.5	6.4	6.6	6.6
B/W pH	6.7	6.5	6.7	6.3	6.8	6.8
B/W Alkalinity, ppm	65.0	61.0	80.0	58.0	91.0	116.0
H/B charge, mv	-60.0					
Chage demand, meq/l	15.0					
FPR	81.0	82.3	80.9	77.1	76.8	79.5
FPAR	42.2	41.2	48.6	44.5	44.8	53.1
Broke						

Flow addition per batch m3	10.0	10.0	10.0	10.0	10.0	10.0
Alcofix kg/MT	0.1	0.1	0.1	0.1	0.1	0.1
Chemicals						
ASA LPM	2.5	2.7	2.6	3.5	5.1	5.5
ASA kg/MT	0.4	0.4	0.4	0.5	0.7	0.6
AKD IVAX 750 LPM						
AKD IVAX 750 kg/MT						
IVAX RTN 100 lpm						
IVAX RTN gm/MT						
IVAX poly TC lpm						
IVAX poly TC g/MT		96.0		97.0		
IVAX Print Jet LPM	0.4	0.3	0.4	0.5	0.5	0.4
IVAX Print Jet kg/MT	2.0	1.8	1.9	2.1	2.3	1.7
PAC LPM	1.1	1.3	1.4	1.1	1.5	1.5
PAC kg/MT	26.6	27.1	28.2	20.2	27.7	24.2
Filler flow LPM	15.0	16.0	21.0	59.0	57.0	69.0
Filler kg/MT	51.0	49.0	62.0	161.0	152.0	160.0
M/C parameters						
Couch vacuum	438.0	450.0	426.0		440.0	437.0
Dryness after couch						
Dryness after press						
Dryness before size press	94.5		93.4			94.5
Dryness final	96.5		96.0			96.2
Steam flow, T/hr	11.3	12.0	12.9	12.6	12.7	14.7
Properties						
Ash%	9.0	8.4	9.5	17.0	18.6	17.1
Cobb, before size press TOP	71.2		68.0			
WIRE	76.1		72.1			
As such cobb TOP	33.4	51.6	32.5	18.8	29.0	33.0
WIRE	38.8	58.5	38.2	54.8	35.6	43.0
Cured cobb TOP	19.3	19.0	19.4	16.0	17.7	20.7
WIRE	21.2	22.3	21.4	18.0	21.6	24.2
cobb after 24hrs TOP	24.7	17.5	18.3	17.8	19.5	21.0
WIRE	29.7	20.2	21.0	20.2	23.0	23.5

Paper properties during the ASA trial at PM-4:Bilt Classic Annexure-7

Properties		Unit	ASA					
DATE			9.4.08	9.04.08	9.04.08	10.04.08	10.04.08	10.04.08
Lot/roll			4848	4855	4849	4853	4852	4850

Substance	NOM	g/m^2	54	60	64	68	70	80
	ACT		55.3	72.5	62.5	67.5	68	80.3
Thickness		micron	77	84	92	88	91	107
Bulk		cc/g	1.39	1.4	1.46	1.43	1.34	1.34
Bust factor			33.2	30.2	29.75	30	21.7	25.2
Tear factor	MD		48.5	53.9	49.75	51.8	48	52.3
	CD		52.6	58.1	53.05	55.6	51	56.2
Breaking length	MD	meter	8354	8005	7582	7794	6500	6706
	CD		4116	3431	3671	3551	3200	3365
Double Fold	MD	no.	31	16	28.5	22	14	27
	CD		16	11	19	15	16	15
As such cobb	Top	gm/m^2	28.8	47.4	22.15	34.8	22.5	37.5
	Wire		33.6	56.9	28.7	42.8	29.7	44.7
Cured Cobb	Top	gm/m^2	18.3	20.5	16.45	18.5	17.5	20.3
	Wire		20.5	25.5	17.65	21.6	20.2	26.9
Wax pick		no.	14	14	14	14	13	13.5
Ash		%	8.7	8.6	9.75	9.2	19	17.3
Gurley porosity		sec/100 ml	13.6	25.3	13.7	19.5	21.2	16.2
Brightness	ISO	%	88.7	89.1	88.9	89	89.2	89.1
Opacity		%	85.3	87.2	88.2	87.7	91.7	92.9
Yellowness		%	-13.6	-13.8	-13.65	-13.7	-13.8	-13.5
Fluorescence		%	19	20.3	18.8	19.5	18.2	18.3
CIE whiteness		%	145.2	146.7	144.4	145.6	146.1	144.65
Smoothness	Top	ml/min	60-140	60-120	50-160	60-120	60-110	60-180
	Wire		120-240	100-240	80-240	130-240	130-190	80-240

9.5 ASA+AKD

To stop hydrolysis of ASA in back water and broke pulp, ASA with AKD was again started. ASA and AKD were added at the dosage of 0.25-0.35 kg/T and AKD 2-4 kg/T respectively. During this period, Print jet at size press was kept 2.0 -2.4 kg/T. Back water pH was in the range of 6.5-7.1. PAC consumption was 20-

40 kg/T. Head box charge was -20 to -50 mv with 10-15 meq/l charge demand. In SS ptg, average FPR and FPAR was 75% and 40%, 79% and 44% , 79% and 42% in 60gsm, 66gsm and 70gsm respectively. And in Bilt Classic, average FPR and FPAR was 79% and 39%, 78.5 and 41% , 79% and 41% in 54gsm, 60gsm and 64gsm respectively. Cobb before size press varied from 75 to 98g/m^2 with cured Cobb of 20-50g/m^2(as such Cobb at pope reel was 20-70 g/m^2). This combination was run from 7.0AM of 11.04.08 to 9.0AM 22.04.08 (11 days). All grammages of Bilt Classic and super ptg were manufactured. About 900 MT was paper manufactured. The major observations during this period were:

- Cleaning frequency of press and lump breaker roll increased which resulted in production loss.
- Some time when back water pH reached at 7.0, picking at press and coating at size press was observed.
- Variation in back water pH was observed.
- Mostly, cured Cobb was in the range of 18-25 g/m^2.

Wet-end data of super ptg and Bilt Classic are given Annexure -3 and 4 respectively. Grammage wise paper properties of Bilt Classic and super ptg are given in Annexure -7 and 8. Temp. dryer profile are attached in Annexure -5.

Avg Wet-end and machine parameters during the ASA+AKD

in Super ptg at PM-4

Annexure-3

Date	13-18.04.08	12,13,16,17.04.08	17,18.04.08
M/c speed, mpm	340	340	340
GSM	60	66	70
Quality	SSP	SSP	SSP
Machine Draw, MT/hr	3.85	4.2	4.49
PULP			
PH	6.5	6.6	6.6
Brightness			
o SR after refining	32.9	29.9	33
o SR M/c chest	34.9	36.3	36
o SR H/B	50.7	46.9	48
H/B pH	6.48	6.42	6.4
B/W pH	6.56	6.6	6.55
B/W Alkalinity, ppm	61	55	58
H/B charge, mv			
Chage demand, meq/l			
FPR	75.1	78.7	78.9

FPAR	39.5	44.0	41.9
Broke			
Flow addition per batch m3	8	8	8
Alcofix kg/MT	0.1	0.1	0.1
Chemicals			
ASA LPM	1.64	1.74	2.58
ASA kg/MT	0.26	0.25	0.34
AKD IVAX 750 LPM	0.5	0.61	0.53
AKD IVAX 750 kg/MT	3.92	4.59	3.53
IVAX RTN 100 lpm			
IVAX RTN gm/MT			
IVAX poly TC lpm			
IVAX poly TC g/MT			
IVAX Print Jet LPM	0.39	0.45	0.45
IVAX Print Jet kg/MT	2	2.12	2
PAC LPM	1.21	1.15	1.53
PAC kg/MT	26	22.8	28.9
Filler flow LPM	38.6	40.7	49.2
Filler kg/MT	120.4	118	134
M/C parameters			
Couch vacuum	443	460	433
Dryness after couch			
Dryness after press			
Dryness before size press	94.3	94.4	94.6
Dryness final	96.4	96.3	96.3
Steam flow, T/hr	12.6	13.5	14.6
Properties			
Ash%	14.5	14	15.6
Cobb, before size press TOP	96.1	67.5	90.4
WIRE	98.2	71.3	91.3
As such cobb TOP	65.7	68.1	70.4
WIRE	70.1	72.5	76.7
Cured cobb TOP	19.7	18.7	20.1
WIRE	22.1	21.2	24.2
cobb after 24hrs TOP	20.4	19.1	20
WIRE	22.8	21.5	23.9

Avg Wet-end and machine parameters during the ASA+AKD

in Bilt Classic at PM-4 Annexure-4

Date	18,19,20,21.04.08	19,21.04.08	20,21.04.08
M/c speed, mpm	335	335	340
GSM	54	60	64
Quality	Bilt Classic	Bilt Classic	Bilt Classic
Machine Draw, MT/hr	3.4	3.9	4.1
PULP			
PH	6.6	6.5	6.5
Brightness	88.1	88.3	89
o SR after refining	32	31	33.5

o SR M/c chest	35	36	36.5
o SR H/B	45	46	40
H/B pH	6.3	6.5	6.2
B/W pH	6.5	6.7	6.5
B/W Alkalinity, ppm		56	48
H/B charge, mv			
Chage demand, meq/l			
FPR	79.4	78.5	78.5
FPAR	39.1	41.2	41.3
Broke			
Flow addition per batch m3	10	10	10
Alcofix kg/MT	0.1	0.1	0.1
Chemicals			
ASA LPM	1.47	1.79	2.19
ASA kg/MT	0.24	0.28	0.33
AKD IVAX 750 LPM	0.29	0.38	0.43
AKD IVAX 750 kg/MT	2.5	3	3.27
IVAX RTN 100 lpm			
IVAX RTN gm/MT			
IVAX poly TC lpm			
IVAX poly TC g/MT			
IVAX Print Jet LPM	0.35	0.44	0.5
IVAX Print Jet kg/MT	2.03	2.31	2.5
PAC LPM	1.5	1.6	1.6
PAC kg/MT	35	34.9	32.2
Filler flow LPM	16.5	39.9	40.7
Filler kg/MT	63.5	126	119
M/C parameters			
Couch vacuum	426	432	440
Dryness after couch			
Dryness after press			
Dryness before size press			
Dryness final	96.3	96.5	96.4
Steam flow, T/hr	12.1	12.8	12.5
Properties			
Ash%	9	10.6	13.9
Cobb, before size press TOP		75	
WIRE		82	
As such cobb TOP		54.7	
WIRE		58.2	
Cured cobb TOP	18.9	26.6	20.1

WIRE	22.0	30.8	21.8
cobb after 24hrs TOP	19	22	20
WIRE	21	26	23

Paper properties during the ASA+AKD trial at PM-4:Super ptg

Annexure-8

Properties		**Unit**	ASA+AKD		
DATE			13,14,18.04.08	12,13,16,17.04.08	17,18.04.08
Lot/roll			4860,67	4859,64	4865
Substance	NOM	G/m²	60	66	70
	ACT		58.8	65	69.4
Thickness		Micron	84	93	96
Bulk		cc/g	1.42	1.41	1.38
Bust factor			24.2	26.2	25.1
Tear factor	MD		45.6	47.5	49.1
	CD		50	52	52.4
Breaking length	MD	Meter	7047	7251	6768
	CD		3597	3819	3526
Double Fold	MD	no.	16	16	20
	CD		9	11	11
As such cobb	Top	Gm/m²	66.4	67.9	59.5
	Wire		69.8	72.9	65
Cured Cobb	Top	Gm/m²	19.3	18.6	18.3
	Wire		20.8	20.7	21.7
Wax pick		no.	14	14	13.2
Ash		%	14.3	14.3	15.4
Gurley porosity		sec/100ml	18.3	18.8	16.8
Brightness	ISO	%	89.9	89.9	89.9
Opacity		%	86.4	87.9	88.4
Yellowness		%	-11.4	-11.4	-11.3
Fluorescence		%	19.2	19	18.9
CIE whiteness		%	138.5	138.3	138
Smoothness	Top	ml/min	60-140	60-180	60-160
	Wire		100-260	120-280	120-300

Tem. Dryer profile at PM -4

Annexure-5

	Blank	Trial			
Date	28.03.08	3.04.08	3.04.08	7.04.08	9.04.08
Time	10.0AM	10.0AM	7.0 PM	9.0 PM	9.0 AM
Quality	Bilt Classic	Bilt Classic	Bilt Classic	Bilt Classic	Bilt Classic
GSm	64	60	70	64	64
0	38	38	40	40	38
1	81	81	80	82	72
2	56	55	79	74	78
3	80	81	81	85	84
4	86	85	73	69	84
5	70	70	71	84	83
6	71	70	53	72	76
7	64	65	63	68	70
8	74	74	74	76	76
9	71	70	74	78	76
10	75	75	74	80	80
11	70	69	70	74	75
12	65	65	57	82	83
13	60	59	68	68	78
14	57	57	71	68	76
15	81	81	70	72	76
16	69	70	65	78	79
17	81	80	83	80	80
18	87	86	81	81	82
19	81	81	85	82	81
20	78	78	78	78	80
21	83	83	82	84	83
22	83	83	78	71	79
23	78	77	84	68	78
Before Size 24	55	53	60	68	80
25	84	84	86	74	70
26	78	78	80	79	80
27	81	81	85	82	83
28	85	85	82	82	82
29	94	94	81	88	85
30	86	86	84	87	86
31	91	92	81	65	82
32	84	83	88	78	78
33	90	91	85	70	72
34	84	84	70	78	76
35	47	48	36	74	73
36	48	48	36	41	40
Steam flow, T/hr	12.3	12.2	13.5	12.7	12.9
Steam pressure, kg/cm²	2.7	2.7	2.7	2.7	2.7
Steam Tem.	140	140	140	142	141

9.6 Observations

1. Machine runnability was normal with the following condition.
   - ASA : Starch ratio 1:1.5
   - ASA 0.25-0.35 kg/T
   - AKD 0.3-4.0 kg/T
   - Print jet 2.0 kg/T
   - Back water pH of 6.5-6.8.
2. Press picking and some breaks at size press were observed when ASA and AKD dosage were more than 0.35 kg/T and 5.0 kg/T respectively.
3. During 100% ASA run, Machine runnability for about one day was normal. After one day, the breaks at size press was observed due to high Cobb before size press i.e. 96 g/m². It indicates that hydrolysis of ASA takes place.
4. When ever FPR and FPAR values increased up to 80+ and 50+ respectively, press picking was observed. To reduce retention, the retention aid was stopped and ASA: Starch ratio was also reduced from 1:2 to 1:1.5.
5. Without any retention aid, FPR and FAPR were also more than the normal run specially in 54gsm.
6. Back water pH was in the range of 6.5 to 7.1. At the pH of 7.0, lump breaker and press picking were observed.
7. Variation in A street pH was observed i.e. 6.3 to 6.8. This has resulted variation in PAC consumption.
8. For the faster curing of AKD, before size press dryers temperature was increased from 56-84 to 68-84°C.
9. Cleaning frequency of press and lump breaker roll was more.
10. There were machine runnability issues mainly in 54gsm.
11. Trial was discontinued due to production loss and poor machine runnability mainly due to chemicals coating at size press and picking at presses.

9.7 Costing

ASA+AKD sizing cost was Rs. 622.0/T of paper against normal neutral sizing Rs. 536.0/MT. This costing is based on the chemicals consumption during the trial i.e. ASA 0.35kg/T, Starch 0.58 kg/T AKD 3.5kg/T, Poly TC 2.4kg/T and PAC 29 kg/T.

9.8 Conclusion:

The practical experience on alkaline sizing was gained using ASA+AKD and ASA alone. The trial could not be continued beyond 15 days as the paper machine got unstable due to press picking problem. Practical problems were observed due to hydrolysis of AKD and ASA, due to re-circulation of white-water which was not the case during laboratory studies.

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