

Developments in Papermaking using Alkaline Sizing in Mix Agro Furnish A Mill Experience

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ABSTRACT

Internal sizing is a widely used process in the paper industry to produce paper grades with hydrophobic character, such as liquid board, lineboard and printing papers. Paper grades with a hydrophobic character have a good resistance to sorption of water. In the acid sizing, a combination of rosin and alum is used. Neutral sizing is usually achieved either with alkyketene dimmer, AKD, or alkenyl succinic anhydride, ASA, AKD and ASA have become more frequently used in the industry during the last decade, not least for economical reasons. Internal sizing at neutral sizing at neutral pH gives the possibility to use calcium carbonate as a filler.

Dispersions of rosin size and AKD can be stored for months, whereas ASA has to be dispersed at the mill due to hydrolysis of ASA. The dispersions can be diluted with water, which facilitates the dosage strategies.

AKD would be sufficient to ensure good paper sizing provided that it forms a chemical bond with furnish. This result is believed to be important in solving some problem associated with AKD sizing and clean backwater system. This paper discuss the approach initiated to establish AKD for improving quality of paper suitable for multicolor printing in mix agro furnish on commercial scale over past two years. The work presented in this article is based on AKD, therefore the rest of the introduction will focus on AKD.

Keyword: Alkaline paper, AKD sizing, retention, size reversion.

INTRODUCTION

Over the last two decades the paper industry has witnessed a rapid conversion from an acid to neutral/alkali system where alkyl ketene dimmer (AKD) is the main sizing agent extensively used in the pH range of 7-10. ⁽¹⁻⁴⁾ AKD's are waxy material which are insoluble in water and have a melting point around 50°C depending on chain length. Commercial AKD's are prepared from natural fatty acid sources and stearic acid is mainly used for this purpose. Industrial AKD's might have a different side chain lengths due to the fact that the source material itself could have a variety of chain lengths ⁽⁵⁾.

Ongoing trends in the paper industry are to minimize the fresh water consumption and to increase the usage of recycled fibers. This can lead to increasing levels of dissolved and colloidal substances in the white water systems. These substances could impair the AKD retention, which consequently can lead to a higher amount of AKD circulating in the wet end system. A higher degree of non-retained AKD could cause problems with agglomeration. Agglomeration could

possibly impair the sizing efficiency, since agglomerates would probably be unevenly distributed on to the fibre material. AKD agglomerates could also cause problems with scaling and spots in the paper.

In the mixing box of the paper machine, a relatively low level of AKD (0.1-0.5% on oven dry furnish) compared to the traditional rosin sizing (2-4% on oven dry furnish) is added to this during internal sizing application. Therefore, maintaining high machine retention is the first important step in order to have better sizing. It is known that AKD retention is quite sensitive to stock pH and it has been reported to be better at pH 8 ⁽⁶⁾.

The second important step in AKD sizing is the reaction mechanism with furnishes, though this is still a point of discussion. In general it is believed that AKD reacts with cellulose fibre and forms a beta- keto ester bond. Hence making paper hydrophobic ⁽⁸⁾. AKD also reacts with water molecules producing an unstable beta-keto acid which then decarboxylates to form a ketone ⁽⁸⁾. The reaction rate between AKD and water is reported to be faster than that with cellulose fibre. There

have been different explanations for the sizing effects of these reaction products. Some researchers claim that the reaction between AKD and cellulose is essential for paper water repellency ⁽⁹⁾. Whereas other believe that such a reaction does not take place at all and is not necessary for sizing ⁽¹⁰⁻¹¹⁾. Calcium carbonate loading is reported to increase AKD demand in paper. However the reason remains to be explained. Therefore it is important to clarify the point to have a better understanding of not only the sizing mechanism, but also the reasons responsible for some problems such as size reversion and lower friction, experienced, when using AKD size paper products. The objectives of this study were as follows:

1. To better understand the retention mechanism of AKD at the wet end.
2. To Study the sizing efficiency and retention using Flocculated AKD dispersion.
3. To study charge demand while using AKD.
4. To study of raw material, chemical consumption at various section and strength properties of pulp and paper while using AKD.

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Material and Method

Pulping and Bleaching

Furnish consists mainly Wheat straw 70.2% ,wood chips about 5.0% and wood pulp 2.0% and Un bleach pulp 1.00% alongwith Talcum -90% is 8.2 %, Talcum -95 is 3.10% and GCC 10.5%. As shown in Table 1.

Pulping & bleaching chemical consumption, cooking & bleaching conditions, and strength properties of hypo pulp is recorded on Table -2 (a) &(b)

Reaction between AKD and Furnish

It is clearly seen that in wet paper there was a negligible amount of reacted AKD (bound AKD), which confirms earlier findings⁽¹²⁾. The small amounts of bound AKD found in wet paper samples could be attributed to two factors. One is the possibility of unstabilized AKD molecules present in the emulsion which may have formed a chemical attachment to the cellulose fibre and filler particles. The second factor is the possibility of dissolved AKD molecules trapped in the inner part of the cellulose fibres and filler cavities. These two factors are equally valid in the evaluation of dry paper extractions.

Experimental techniques

a) Britt Dynamic Drainage Jar:

The Britt Dynamic Drainage Jar was used in the experimental setup for studying the colloidal stability of the AKD⁽¹³⁾ dispersions and used for pre-flocculation. The AKD dispersions were added to synthetic and real white water samples in the jar and stirred at speeds ranging from 1000-2000 rpm with varying dose of polyelectrolyte.

b) Polyelectrolyte titration: The polyelectrolyte titration were performed in streaming current mode⁽¹⁴⁾.

The function of the streaming current detector is to create a high flow rate in the sample in order to separate the counterions from the polyelectrolytes attached to the piston or the walls of the sample vessel. A piston closely fit to the narrow cylindrical section in the sample

Table: 1 Furnish during AKD Trial

	Furnish						
	Wood Chips	Wheat straw	Unb. Pulp	Wood Pulp	Talcum %		GCC 94
					90	95	
Min	1.0	62.8	0.70	1.8	6.2	1.6	5.0
Max	6.2	74.7	2.00	2.8	9.8	4.4	10
Avg	5.6	70.2	1.00	2.0	8.2	3.10	10.5

Table :2 (a) Chemical consumption in various section of pulping and bleaching

	Cooking Chemical			Cooking Condition			Bleaching Chemical		
	NaOH		Pulp Aid	Time (min)	Temp °C	Press. Kg/cm ²	Cl ₂ %	NaOH Ext. %	H ₂ O ₂ %
	Agro	W/C							
Min	15.4	19.54	0.03	21.4	162.4	7.18	8.91	2.62	0.62
Max	16.08	20.23	0.06	22.4	167.0	7.50	9.89	2.97	1.09
Avg.	15.6	19.9	0.05	22.1	164.1	7.28	9.35	2.74	0.94

Table-2 (b) Strength properties of Hypo pulp sheets

Hypo Pulp Properties				
	Breaking Length	Burst Factor	Tear Factor	Brightness %
Min	3015	16.9	57.5	77.8
Max	3555	22.8	61.2	79.6
Avg.	3420	20.1	58.25	78.6

Table:3 Properties of the AKD dispersion

Dispersion	Stabilizing Polymer	Charge density of polymer	Solids content % w/w	AKD Content % w/w
A	Cationic starch	Low	10	7.7
B with PA	Cationic starch (Polyaluminum salt)	Medium	13.3	10
C	Polyamideamine	High	10	8.9

vessel creates a high flow in the sample. The streaming current is created when the counterions are separated from the polymers. The sample vessel contains two electrodes that collect the streaming current and convert it to the corresponding potential. A polyelectrolyte with opposite charge is added in portion to the sample until the potential reaches zero (end point of the titration). The polyelectrolyte titrations with streaming current detection were made with a Mutek PCD 03 pH, Mutek Analytical, Germany.

The charge density of AKD-density of AKD dispersions was analyzed using streaming current detection and the cationic demand of the white water samples was analyzed using photometric detection

Characteristic of AKD dispersion

Three dispersion with different composition were chosen to cover product groups on the market. The dispersion were made from the same AKD wax, with a melting point of 52-

53°C and the average size of the AKD particles was approximately the same, 0.5-0.8 µm in diameter. The only difference between the dispersion was the surface charge, attained by using different stabilizing polymer. The stabilizing polymer, the solids content and the AKD Content of the dispersion are presentation in Table-3.

Machine Chemicals

AKD Consumption, charge analysis and retention are recorded in Table -4,5 & 6 during trial of AKD Since AKD consumption is comparatively high due to close loop of back water system more deposit build up observed which resulted in sheet break on paper machine.

Problem encountered

Excess breakage on machine due to holes, fibre loss & slime etc, reduced boilout frequency to 15-20 days against, 30-35 days in neutral sizing earlier. Defected samples were drawn and sent to FT-IR analysis.

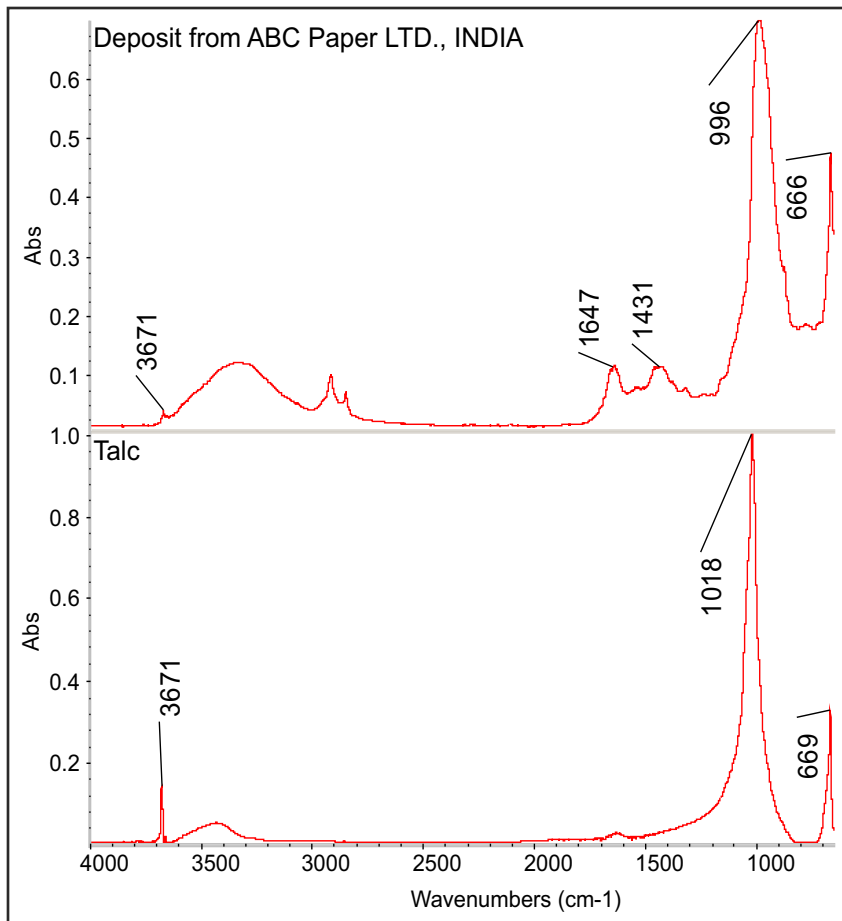
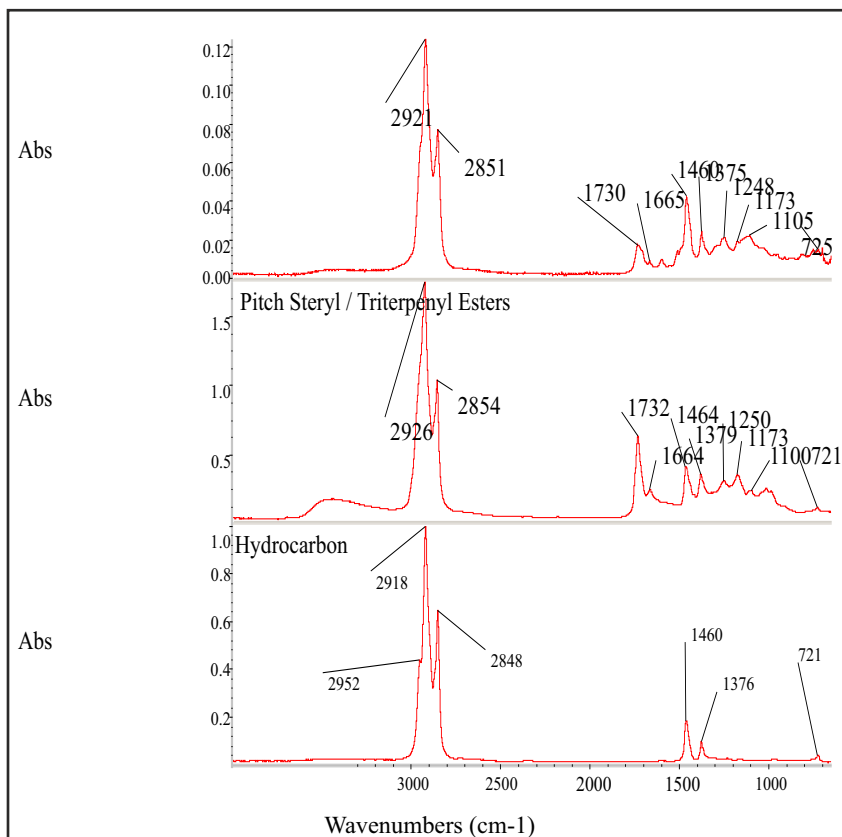


Figure 1. FT-IR spectra of the deposit and a reference spectrum of talc. The presence of talc was readily observable in the deposits based on the presence of characteristic peaks at 3671, 1018 and 669 cm^{-1} (talc).



FT-IR analysis

Based on the FT-IR analysis, the deposit was found contain of talc, hydrocarbon oil and traces of pitch/wood pitch

The deposit and sheet defect may be caused by hydrocarbon oil and pitch in the furnish.

The sample was analyzed using microscopy and FT-IR spectroscopy. The following reasons were identified for the sheet break on machine. Details report is shown Fig. 1&2.

Strength properties of paper during AKD trail is recorded in Table-7

RESULTS AND DISCUSSION:

Table-1 shows the furnish which are used in AKD trial wheat straw 70.2%, woodchips 5.0% and wood pulp 2.0%, Taclum-90 & 95 are used 8.2 & 3.10% GCC 94 10.5%.

Table 2(a) shows the average cooking chemicals consumption in agro & wood chips.i.e 15.6 &19.9% NaOH.& Pulp aid 0.05%. Cooking time is 22.1 min. Temperature is 164 and Pressure is 7.28 kg/cm^2 . In bleaching (C-E_p-H-H sequence) average chlorine consumption is 9.35%, Average NaOH consumption in alkali extraction is 2.74%. and hydrogen peroxide is 0.94%.

Table -2 (b) shows the strength properties of Hypo pulp .average breaking length, burst factor 3420 & 20.1 and tear factor and brightness of hypo pulp is 58.52 & 78.6.

Table: 3 shows the properties of dispersion. Dispersions A and B with PA were stabilized with cationic starch, which also provides them with steric stabilization. Dispersion A had the lowest charge density. B with PA had a medium charge density and also contained a polyaluminum salt (PA). Dispersion C had the highest charge density and was stabilized with cationic polyamideamine. Dispersion C should be less sterically stabilized than the other two.

Fig 1 & 2 show that based on the FT-IR analysis, *the deposit* was found to contain talc, hydrocarbon oil and traces of pitch/wood pitch.

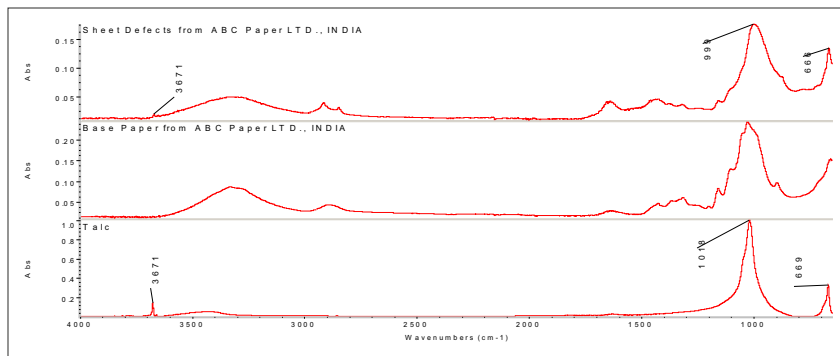


Figure 3. FT-IR spectra of the sheet defect and a reference spectrum of talc. The presence of talc was readily observable in the sheet defect based on the presence of characteristic peaks at 3671, 1018 and 669 cm^{-1} (talc).

Table : 4 Machine chemicals during AKD Trail

Dt	AKD (Kg/T)	BIOCIDE			Retention Aid	ATC (g/T)	FPR	FPAR
		A	B	C				
01	18.0	11.2	22.5	12.4	78.8	150.0	77.1	46.1
02	17.6	11.4	23.7	14.0	79.3	150.0	76.2	46.0
03	17.1	12.4	25.7	15.7	85.7	150.0	75.9	43.9
04	18.2	11.2	23.3	14.3	60.5	150.0	79.3	47.4
05	17.7	11.0	22.8	14.3	59.0	150.0	77.6	46.8
06	19.8	11.0	22.0	12.1	60.7	150.0	75.8	45.9
07	19.1	10.7	22.3	14.0	58.0	150.0	75.4	44.8
08	19.2	10.0	20.7	13.0	65.3	150.0	76.2	45.3
09	20.9	10.0	21.0	13.2	65.8	150.0	77.06	44.2
10	22.8	11.0	22.8	14.3	63.3	160.0	77.6	43.7
11	22.3	11.6	24.1	15.2	80.4	160.0	77.8	44.9
12	21.9	11.8	24.6	15.5	77.5	160.0	77.0	46.2
13	21.0	11.8	24.6	15.5	73.0	180.0	75.6	46.8
14	21.3	12.0	25.0	15.7	74.0	180.0	76.6	47.2
15	19.4	11.0	22.8	14.4	67.6	180.0	76.2	47.1
16	20.6	10.3	25.8	15.5	67.2	200.0	77.0	46.2
17	22.7	12.8	26.7	16.8	69.2	200.0	78.3	47.9
18	20.4	12.0	25.0	15.7	90.0	250.0	76.7	46.8
19	19.6	11.1	23.1	14.5	120.0	250.0	77.1	46.5
20	19.9	10.4	21.5	13.5	150.0	250.0	77.8	47.1
21	19.2	10.6	22.1	13.9	180.0	250.0	78.6	47.9
Min	17.1	10.0	20.7	12.4	58.0	150.00	75.4	44.2
Max	22.8	12.8	26.7	16.8	180	250.0	79.3	47.9
Avg.	19.9	11.20	23.43	14.45	82.15	179.52	76.99	46.12

Table: 5 Charge Analysis during AKD Trail

DATE	TIME	FINAL PULP		HEAD BOX No. 1		HEADBOX No. 2		HEAD BOX No. 3	
		CHARGE (mV)	DEMAND (meq/ltr)	CHARGE (mV)	DEMAND (meq/ltr)	CHARGE (mV)	DEMAND (meq/ltr)	CHARGE (mV)	DEMAND (meq/ltr)
03	16.30	-180	200	-170	100	-160	90	-160	80
04	14.30	-190	100	-180	90	-165	85	-170	75
05	14.45	-205	115	-200	74	-195	75	-250	43
06	10.45	-190	170	-140	105	-145	105	-160	100
07	11.30	-137	218	-170	138	-160	112	-160	115
08	11.45	-150	145	-170	110	-160	115	-145	126
09	09.45	-145	187	-195	98	-189	94	-190	95
10	11.30	-150	200	-190	100	-190	105	-170	80
11	12.30	-190	171	-167	107	-187	102	-170	88
12	17.30	-105	205	-190	145	-182	140	-175	165
13	10.30	-125	190	-190	140	-195	145	-150	130
14	11.30	-120	210	-170	145	-165	140	-170	125
15	11.30	-150	220	-180	135	-175	130	-175	135
16	10.30	-180	210	-180	140	-180	140	-150	120
17	16.30	-140	225	-145	135	-160	140	-145	135
18	15.30	-150	250	-160	150	-175	170	-160	190
19	10.30	-140	300	-170	200	-165	210	-150	180
20	10.45	-100	480	-160	253	-160	250	-140	366
21	11.30	-100	490	-120	332	-115	310	-105	292
Min.		-205	100	-200	74	-195	75	-250	43
Max		-100	490	-120	332	-115	310	-105	366
Avg.		-149.84	225.27	-170.89	141.94	-169.63	139.89	-162.89	138.94

Spectroscopic Analysis of Deposit

The deposit was analyzed using FT-IR spectroscopy. An infrared spectrum of the deposit was acquired and is shown in Fig 2 along with a reference spectrum of talc

Figure 2. FT-IR spectra of the sheet defects extractables along with a reference spectrum of wood pitch steryl/triterpenyl esters and hydrocarbon. The presence of pitch steryl/triterpenyl esters and hydrocarbon were readily observable in the sheet defects extractables based on the presence of characteristic peaks at 2926, 2854, 1732, 1664, 1464, 1379, 1250, 1173, 1100 and 721 cm^{-1} (wood pitch steryl/triterpenyl esters), 2952, 2918, 2848, 1460, 1376 and 721 cm^{-1} (hydrocarbon oil).

Spectroscopic Analysis of Sheet Defect

The sheet defect was analyzed using FT-IR spectroscopy. An infrared spectrum of the sheet defect was acquired and is shown at Fig 3 along with a reference spectrum of talc.

Table: 6 Average Retention at various gsm of paper

Quality & GSM		H.Box Cy%	B.Water Cy%	Stuff Box Cy%	H.Box ⁰ SR	F.P.R
ABC Gold -58	Min	0.72	0.17	2.84	49	74.36
	Max	0.98	0.22	3.01	52	78.72
	Avg.	0.80	0.19	2.91	47.77	76.58
ABC Gold-54	Min	0.71	0.14	2.5	50	74.39
	Max	0.88	0.22	3.02	51	81.25
	Avg.	0.76	0.18	2.82	45.40	77.20
ABC Gold-60	Min	0.70	0.16	2.84	49	73.56
	Max	1.05	0.23	3.06	52	78.31
	Avg.	0.85	0.20	2.97	50.27	76.79
ABC Gold- 64	Min	0.88	0.20	2.86	48	74.47
	Max	0.97	0.24	3.10	51	77.27
	Avg.	0.93	0.22	3.00	49.83	76.22
ABC Gold-68	Min	0.98	0.22	2.88	48	77.55
	Max	1.00	0.22	3.12	50	78.0
	Avg.	0.99	0.22	3.00	49.0	77.78
ABC Gold-70	Min	0.94	0.21	2.80	48	76.04
	Max	1.03	0.23	3.16	52	79.61
	Avg.	1.00	0.22	2.92	49.64	77.55
ABC Gold-80	Min	0.98	0.20	2.56	49	76.34
	Max	1.06	0.24	3.20	51	79.81
	Avg.	1.00	0.22	3.01	50.25	78.21
ABC Gold-90	Min	1.06	0.23	3.12	50	77.27
	Max	1.10	0.25	3.20	51	78.3
	Avg.	1.08	0.24	3.16	50.5	77.79

Table: 4 shows the average AKD consumption i.e 20.08 Kg/T Biocide A, B and C are 11.31,23.6 and 14.6 and retention aid 8215. ATC 179.52. FPR 76.99 FPAR 46.12 during AKD trial. Due to excess deposit build up in the system, fixative and ATC dose increased to maintain the charge and better bonding of fibre and filler. However First Pass Retention maintained below 80% to ensure smooth runnability of paper machine.

Table 5 shows charge analysis-Charge demand of whole system in different paper quality has been studied throughout the month. The chart clearly indicates charge demand readings were

steady for whole month, but at the month end charge demand readings suddenly shoot up. It might be due to quality change which may result in highest AKD consumption during those days.

Table: 6 shows the first pass retention, head box cy%, back water cy% stuff box cy% head box OSR at various gsm of paper

Table :7 Shows the strength properties of paper in various gsm

CONCLUSIONS

By shifting neutral sizing to alkaline sizing tremendous improvement

noticed in paper smoothness, dimensional stability, reduction in fluff and creasing of paper. In mix agro furnish it was found that in general the reacted part of sizing chemical was quite small compared with total retained AKD. The rest of the unreacted AKD and those adsorbed on to fillers are of great concern since it is thought they cause a size reversion as a result of hydrolyzation in time. Therefore , it is concluded that the use of an appropriate amount of AKD in paper should be applied as well as developing some quick way to make them all chemically attached to the furnishes to get a perfect sizing operation. As reacted portion of added AKD was so small that excessive size addition should be avoided by promoting an increase in the quantity of reacted AKD in paper. Reduced AKD consumption will ensure less deposits buildup, clean back water system and increased boil-out frequency.

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Table: 7. Strength Properties of paper at various gsm

Dt	Qty & GSM	Ash %	Burst Factor	Breaking Length		Tear Factor		Cobb		Smoothness	Br %	Whiteness
				MD	CD	MD	CD	T/S	W/S			
01	ABC Gold- 70	21.1	17.4	3990	2416	38.1	41.0	25.2	26.5	62/115	79.4	137.0
02	ABC Gold- 58	21.4	19.0	4412	2567	38.1	41.6	25.0	26.0	80/170	76.3	136.0
03	ABC Gold- 54	15.5	20.1	4556	2745	38.8	42.5	25.5	26.5	70/120	78.4	134.0
04	ABC Gold- 54	15.6	19.5	4359	2536	41.3	44.3	25.0	26.0	75/120	78.1	138.0
05	ABC Gold- 80	22.2	16.8	4266	2454	38.3	40.9	25.7	26.8	72/100	79.1	1400.0
06	ABC Gold- 60	19.8	18.4	4065	2499	38.7	42.1	25.8	26.9	60/110	78.4	139.0
07	ABC Gold- 64	20.4	17.7	4584	2534	38.7	41.9	25.0	26.0	70/130	79.1	137.0
08	ABC Gold- 68	20.1	18.5	4297	2516	38.8	41.8	25.6	26.8	65/105	78.9	138.0
09	ABC Gold- 70	22.2	17.4	4132	2402	38.6	41.5	25.0	26.0	80/160	78.6	139.0
10	ABC Gold- 60	19.5	18.5	4019	2473	40.3	43.6	25.7	26.9	70/150	78.8	142.0
11	ABC Gold- 58	19.0	18.9	4380	2589	39.5	42.9	25.3	26.4	70/110	78.5	141.0
12	ABC Gold- 60	20.6	19.1	4297	2507	39.4	42.7	25.8	26.8	65/120	78.8	143.0
13	ABC Gold- 58	19.9	18.7	4271	2441	39.7	43.2	25.0	26.0	60/115	78.8	140.0
14	ABC Gold- 58	20.1	19.2	4330	2454	38.3	41.8	25.8	26.9	75/130	78.3	139.0
15	ABC Gold- 70	21.3	17.7	4493	2607	39.8	42.7	25.2	26.5	65/110	79.3	143.0
16	ABC Gold- 80	21.8	16.0	4114	2502	39.9	42.5	25.0	26.2	80/115	78.8	140.0
17	ABC Gold- 54	16.2	20.1	4538	2719	39.5	43.2	25.1	26.4	75/110	78.6	137.0
18	ABC Gold- 54	14.8	18.7	4567	2786	40.3	43.5	25.4	26.8	65/120	78.7	140.0
19	ABC Gold- 70	22.7	18.6	4421	2608	38.1	41.1	25.8	26.9	65/100	78.5	139.0
20	ABC Gold- 80	23.3	16.7	4221	2411	38.6	41.6	25.0	26.0	70/110	79.1	140.0
21	ABC Gold- 70	21.6	18.1	3431	2570	40.2	43.1	25.4	26.6	80/115	79.1	137.0
22	ABC Gold- 64	20.2	19.2	4706	2550	40.6	43.8	25.5	26.5	80/110	78.1	140.0
23	ABC Gold- 58	20.8	18.1	4150	2566	40.3	43.7	25.0	26.0	65/110	79.2	137.0
24	ABC Gold- 58	19.8	18.6	4252	2437	39.1	42.7	25.2	26.3	70/120	78.4	138.0

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