Impact of recent pulp-mill modifications on sulfur contents in crude sulfate turpentine

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ABSTRACT: The average sulfur content of crude sulfate turpentine (kraft) received at the Union Camp–BBA terpene and aromatics plant has increased from 4400 ppm in 1989 to 7000 ppm in 1993. Comparison of operating conditions and turpentine sulfur contents at several mills reveals strategies for reducing sulfur in turpentine. For hot-blow batch digesters, simple adjustments in digester steaming should reduce sulfur to 25–30% of current levels without reducing turpentine yield. For continuous digesters, increased presteaming of the chip bin and higher decanter operating temperatures should reduce sulfur somewhat. Recycling of condensate-stripper foul (red) oil to the turpentine decanter must be avoided. Foul oil contains high concentrations of reduced-sulfur compounds that are very difficult to remove and have a highly offensive odor.

KEYWORDS: Batch digesters, chemical composition, continuous digesters, kraft mills, operating conditions, sulfate turpentine, sulfur compounds, turpentine, vertical design.

Annually, 40–45 million gallons of crude sulfate turpentine (CST) are recovered in North America. In recent years, many of the mills that supply kraft CST to fractionation plants have modified their pulping processes to comply with air-quality and odor-emission standards. One consequence has been an increase in the sulfur content of CST. Kraft turpentine has become extremely foul smelling, presenting significant difficulties to the turpentine processor.

The yield of turpentine per ton of pine wood has increased at many mills, with an accompanying increase in the sulfur content of their kraft turpentine. In 1989, the average sulfur content of CST supplied to our terpene and aromatics plant (Union Camp–BBA, Jacksonville, FL) averaged 4400 ppm sulfur. In 1993, the average sulfur content of CST had risen to 7000 ppm sulfur. Some mills are supplying turpentine containing as much as 40,000 ppm sulfur. At our facility, a sulfur content of less than 6000 ppm is considered acceptable.

The sulfur in the turpentine consists largely of organic reduced-sulfur compounds. Of particular concern is the fact that the species of organic reduced-sulfur compounds in the kraft turpentine has been shifting to those that are much more difficult to remove. Removal and destruction of these reduced-sulfur compounds represent an increased environmental-emissions concern. Studies indicate that differences in operating technique at the mill sites can greatly reduce the sulfur content of turpentine.

At our facility, four mills supplying a small portion (21%) of the overall turpentine supply contribute more than 53% of the sulfur input. The principal contributors to the sulfur load at our facility are listed in **Table I**.

Hot-blow batch digesters

Our investigation into how to reduce the sulfur content of CST focused initially on mills using hot-blow batch digesters. CST from hot-blow batch

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digesters represents about 65% of our total supply. About 40% of our turpentine supply comes from hot-blow batch digesters producing pulp for bleaching. These mills represent about 46% of the sulfur input. The other 25% of the turpentine from hot-blow batch digesters comes from mills producing unbleached pulp. White-liquor sulfidities are 32–36% at the bleached mills and 25–28% at the unbleached mills.

Mill comparisons

Table II shows the operating data for the hot-blow batch mills supplying CST to our facility, including the two high-sulfur-input mills listed in Table I (Mills A and D). The data for Mills B and E show that hot-blow batch mills can produce CST at high yields while maintaining sulfur content at acceptable levels under 6000 ppm.

Bleached kraft mills

Mill B produces bleached kraft pulp at 32–34% sulfidity. Turpentine from Mill B averages about 3700 ppm sulfur. The pulping process and grade structure at Mill B compare well with those of Mills A and C, which produce bleached pulp at 32–36% sulfidity. The critical difference between Mill B and Mills A and C is that Mill B does not recycle the secondary blow-heat condensate to the turpentine decanter. Instead, the mill recycles the condensate to the washers. The mill can do this because the condensate has negligible turpentine content and relatively low odor. Nonetheless, Mill B enjoys excellent turpentine recovery of about 4.04 L (0.97 gal)/o.d. (oven dry) metric ton of pine wood charged to the digester.

Figure 1 shows the turpentine yield and the sulfur content for Mill A from 1989–93. **Figure 2** shows comparable statistics for Mill B. Before startup of a blow-heat recovery system at Mill A, the sulfur content of the turpentine produced during the cook averaged 3650 ppm. Since that startup, the overall sulfur content of the turpentine has averaged 12,600 ppm. The sulfur content of the blow-heat turpentine at Mill A is calculated to be 28,500 ppm. This high sulfur content of the blow-heat turpentine was confirmed indirectly at Mill C, which can easily isolate blow-heat turpentine from turpentine produced during the cook. Mill C measured the cook turpentine at 5100 ppm sulfur and the blow-heat turpentine at 26,500 ppm.

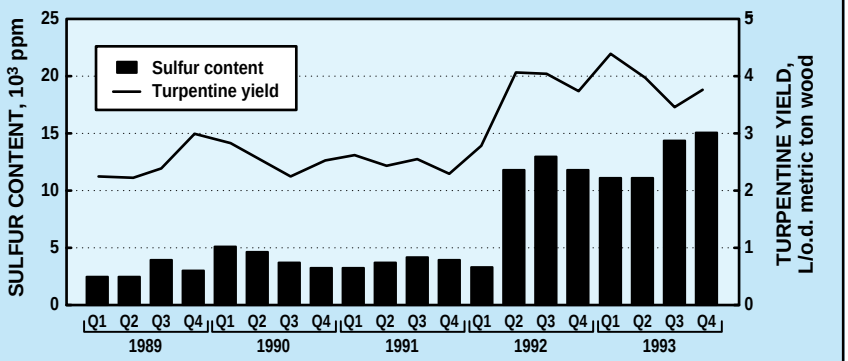
The observation that blow-heat turpentine contains more sulfur than digester turpentine is logical. At cooking temperature, the short-chain hydrocarbons produced during delignification react with the sulfide ions present in the cooking liquor. The resulting reduced-sulfur organic compounds are only partially vented during the time at cooking temperature, but they are more fully released when the liquor in the blown pulp flashes. When the flashed steam is condensed, the turpentine also condenses. Since turpentine is an excellent organic solvent, these sulfur-containing organics are concentrated in the condensed turpentine. The data indicate that the reduced-sulfur compounds saturate

I. Principal contributors to sulfur load

Supplier	*Turpentine supply, %*	*Sulfur input,%*	*Comments*
Bleached kraft			
Mill A	13	25	Hot-blow batch digester, blow-heat condensate to turpentine decanter
Mill F	2	10	Cold-blow continuous digester [a]
Mill G	2	10	Cold-blow continuous digester [b]
Unbleached kraft			
Mill D	4	8	Hot-blow batch digester, blow-heat condensate to turpentine decanter
Total	21	53	

[a] Dual-vessel Kamyr MCC (modified continuous cooking) digester
[b] Single-vessel Kamyr MCC digester

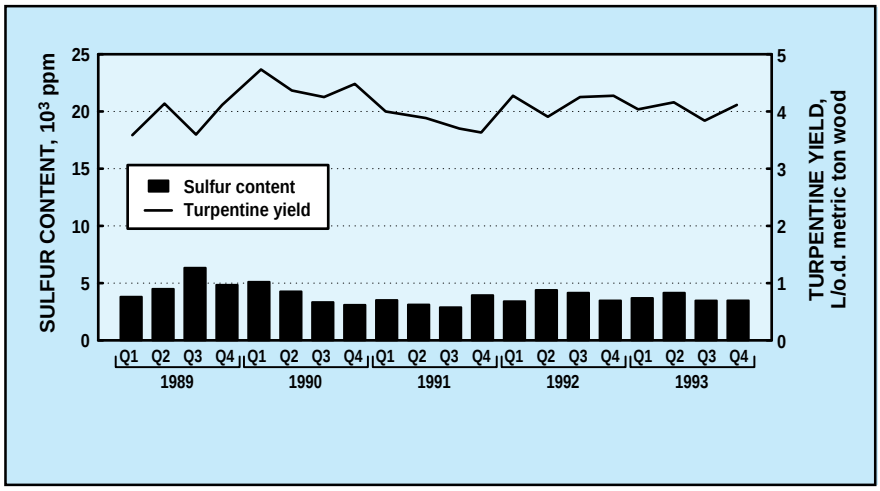
1. Turpentine yield and sulfur content for Mill A (hot-blow batch digester, bleached kraft pulp, blow-heat condensate to turpentine decanter)



II. Sulfur in turpentine—hot-blow digesters

Supplier	*Sulfur in CST, ppm*	*Turpentine yield, L/metric ton*	*White-liquor sulfidity, %*	*Comments*
Bleached kraft				
Mill A	12,993	3.93	35	Blow-heat condensate to decanter
Mill B	3,726	4.04	33	Blow-heat condensate to showers
Mill C	9,164	3.50	34	Blow-heat condensate to decanter
Unbleached kraft				
Mill D	12,961	4.22	28	Blow-heat condensate to decanter
Mill E	1,154	3.83	27	Blow-heat evaporator condensate to stripper

2. Turpentine yield and sulfur content for Mill B (hot-blow batch digester, bleached kraft pulp, blow-heat condensate to showers)



the blow-heat turpentine upon condensation, as both bleached and unbleached blow-heat turpentine contain about 26,000–28,000 ppm sulfur.

Unbleached kraft mills

The same phenomenon occurs at mills producing unbleached pulp in hot-blow batch digesters. At Mill D, blow-heat recovery is accomplished by a proprietary system that uses the blow heat to strip condensate and operate a rectification column. Before startup of the blow-heat recovery system, turpentine recovery averaged 2.92 L (0.7 gal)/o.d. metric ton of pine wood, and the sulfur content of the turpentine averaged 1300 ppm. Since startup, the turpentine recovery has averaged 4.22 L (1.01 gal)/o.d. metric ton of pine wood, while the overall sulfur content of the turpentine has averaged 13,000 ppm. **Figure 3** shows turpentine yield and sulfur content for Mill D from 1989 to 1993. The blow-heat recovery process collects both blow-heat turpentine and condensate-stripper foul (red) oil. The mixture of blow-heat turpentine and foul oil is calculated to contain 24,300 ppm sulfur. This high sulfur content is consistent with that calculated at Mill A and measured at Mill C.

Much of the sulfur being recycled at Mill D appears to be coming from the rectification-column condensate. The mill has increased the rectification-column overhead temperature from 80°C (175°F) to above 91°C (195°F). This has helped to volatilize much of the methanol and the associated TRS (total reduced sulfur) compounds. The sulfur content of the blow-heat turpentine is now 8000–9000 ppm. Work is underway to complete both a turpentine and sulfur balance around the blow-heat recovery system.

Mill E operates batch digesters with blow-heat recovery. **Figure 4** shows turpentine recovery and sulfur content for Mill E from 1989–93. Turpentine recovery averages about 3.83 L (0.9 gal)/o.d. metric ton of pine, with a sulfur content of about 1150 ppm. Mill E uses the blow heat to preevaporate the weak liquor, similar to the practice at Mill A, where the sulfur content is unacceptably high. However, Mill E does not recycle the blow-heat condensate to the turpentine decanter, similar to the practice at Mill B, where the sulfur content also is low.

Given these results, it is clear that the central issue in reducing turpentine sulfur content involves increasing digester turpentine while virtually eliminating blow-heat turpentine.

Reducing production of blow-heat turpentine

The key to reducing production of blow-heat turpentine is rapid heating of chips to cooking temperature. Sampling at Union Camp's mill in Savannah, GA, and data in the literature *(1, 2)* have shown that much of the digester turpentine is evolved during the ramp to cooking temperature (pressure) before the delignification reaction has begun. This is illustrated in **Fig. 5**.

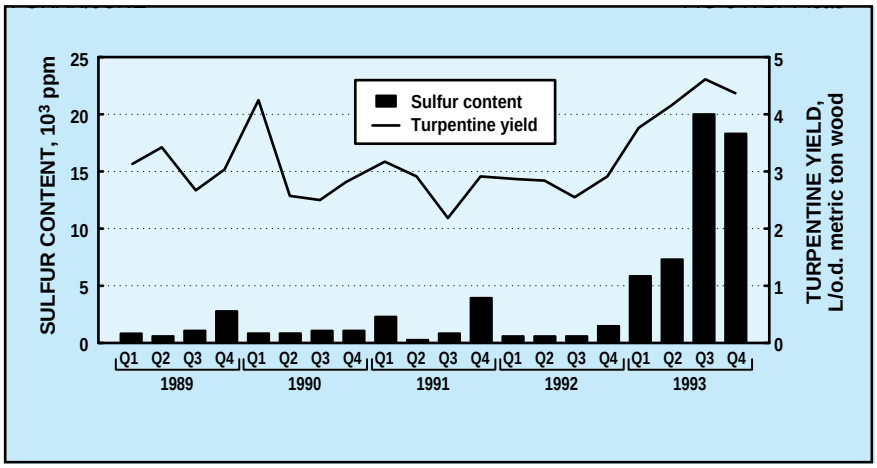
At the time of that study *(1, 2)*—the late 1960s—blow-heat recovery systems were almost nonexistent. Turpentine not vented to the collection system was flashed to the atmosphere when the digesters were blown. Hence mills having low turpentine recovery were not recovering the turpentine remaining in the digester when the pulping reaction began.

At bleached Mill A, the digester steam valves allow a maximum steaming rate of 160 kg/h/m^3 (9–10 lb/h/ft^3) of digester capacity. The steam-valve position is fully open until the desired cooking temperature (pressure) is achieved.

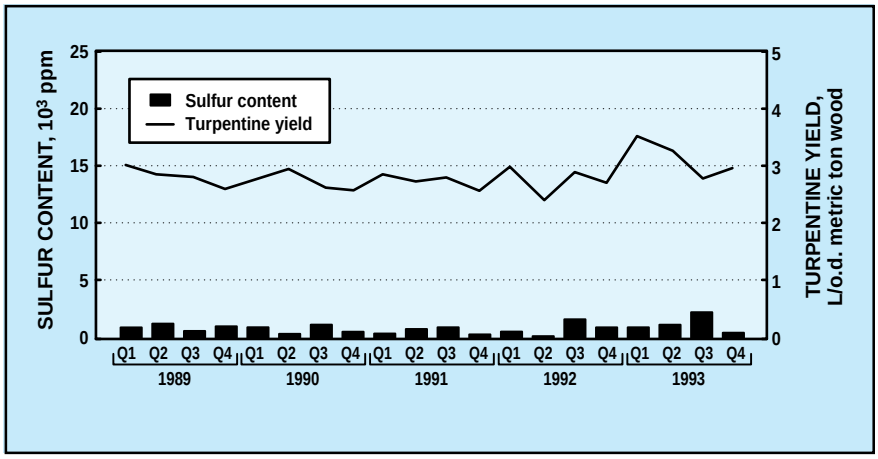
At bleached Mill B, the digester steam valves allow a maximum steaming rate of 230 kg/h/m^3 (14–15 lb/h/ft^3) of digester capacity. At a digester pressure of about 345 kPa (50 psi), the steam-valve opening is reduced to about 80%. At about 585 kPa (85 psi), it is reduced to about 50%. It is finally reduced to less than 20% at cooking temperature, just enough to maintain good digester circulation. **Figure 6** shows the heating profiles for Mills A and B.

Bleached Mill C has a maximum steaming rate of about 162 kg/h/m^3 (10 lb/h/ft^3) of digester capacity. Similarly, unbleached Mill D has a maximum steaming rate of 192 kg/h/m^3 (12 lb/h/ft^3) of digester capacity.

3. Turpentine yield and sulfur content for Mill D (hot-blow batch digester, unbleached kraft pulp, blow-heat condensate to turpentine decanter)



4. Turpentine yield and sulfur content for Mill E (hot-blow batch digester, unbleached kraft pulp, blow heat to preevaporator)



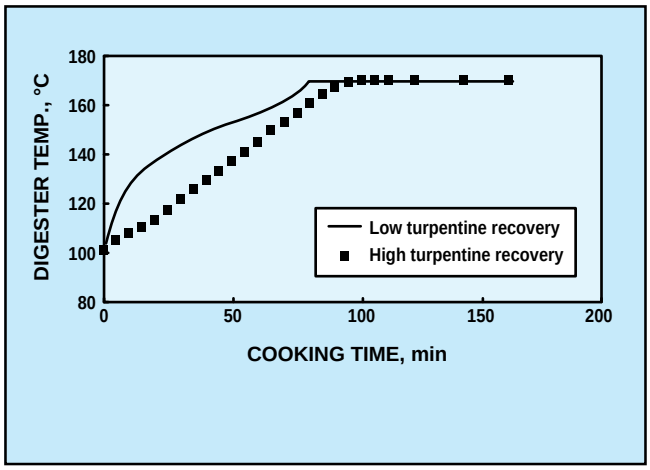
Operational considerations

Two concerns have been raised about increasing the maximum digester steam flow when ramping the digester to cooking temperature.

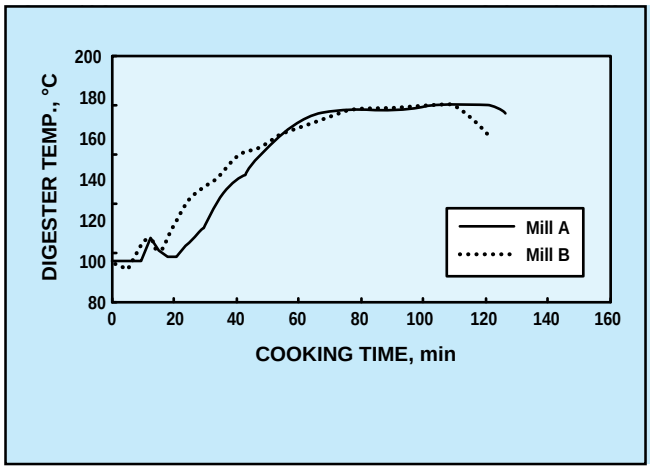
1. Increased digester steam consumption. This does not appear to be a reasonable concern, since the heat required to bring a digester to cooking temperature is determined by the mass of chips, liquor charge, and the time at cooking temperature.
2. Increased variation of digester steam header pressure. Heavier steaming at the start of the cook should not increase variation of digester steam header pressure. This is because at about the time a second digester would begin steaming, the first digester's steaming rate would be reduced. Careful control of the batch cycle times and steaming rates should avoid any such problems.

This strategy not only reduces the sulfur content of the turpentine, but it provides an additional benefit in that the TRS vapors do not condense with the blow-heat condensate, allowing a higher percentage to pass on for incineration. This permits the blow-heat condensate to be reused more widely. Furthermore, there is

5. Temperature profiles for hot-blow batch digesters at two levels of turpentine recovery



6. Temperature profiles for hot-blow batch digesters at Mill A (high sulfur content) and Mill B (low sulfur content)



III. Trial results for modified venting sequence at Mill A

	Sulfur in turpentine			
	Baseline cook		*Modified cook*	
	%	*ppm*	*%*	*ppm*
Turpentine to pressure	18	1,349	36	1,007
Turpentine at pressure	47	10,472	53	10,377
Turpentine in blow gas	35	24,700	11	24,700
Sulfur in turpentine	...	13,809	...	8,579
Total	100		100	

less chance that turpentine will collect and accumulate in the NCG (noncondensible gas) collection system. Turpentine accumulation in NCG systems has been implicated in several fires and explosions.

Batch-digester venting-sequence trial

At Mill A, a trial was conducted in which the digester vent gas was collected, condensed, and decanted for the "ramp to cooking pressure" and the "at cooking pressure" phases. In the baseline portion of the study, the digester ramped to cooking temperature in the normal fashion with the vent valve fully open until cooking pressure was achieved. In the trial cooks, the vent valve was throttled at about 344 kPa (50 psi). The volumes of turpentine collected were noted for each phase, and the sulfur content and organosulfur species were determined on all of the turpentine samples collected. The data for sulfur content are presented in **Table III**. The cooking-pressure profiles for the baseline operating procedure and the modified procedure are illustrated in **Fig. 7**. The trial showed that small adjustments in venting reduced the sulfur content of the turpentine.

Another finding was that turpentine produced during the ramp to cooking pressure contained almost no methyl mercaptan. **Table IV** compares the sulfur species in the overall blend of turpentine from Mill A and Mill B. These results further demonstrate that Mill B recovers most of its turpentine from the ramp to cook pressure.

Continuous digesters and cold-blow batch digesters

About 30% of the turpentine supplied to our facility comes from continuous (Kamyr) digesters. About 7% of the turpentine supply comes from continuous digesters producing bleached pulp, while 23% comes from digesters producing unbleached pulp. The bleached mills represent about 25% of the overall sulfur input, while the unbleached mills represent 15% of the sulfur input.

Mill comparisons

Comparison of mill data from mills using cold-blow processes—both continuous and batch—did not suggest a clear course of action, unlike the case of mills using hot-blow batch digesters. **Table V** summarizes the annual sulfur content and turpentine yield for a broad variety of cold-blow digester processes.

Continuous digesters

Unlike the hot-blow batch digesters, the higher white-liquor sulfidity

at the bleached mills appears to result in much higher sulfur content in their turpentine than at the unbleached mills. If turpentine recovery from the chip bin could be increased by increased fresh steam use, the turpentine should have less sulfur content. The data in Table IV also suggests that reducing liquor carryover and formation of turpentine emulsion may also reduce the sulfur content of the turpentine.

Results of studies at the Union Camp mill in Savannah and Mill G have shown that for digesters with two-stage turpentine condensing, the sulfur compounds concentrate in the secondary-condensate turpentine. The results are shown in **Table VI**.

Unfortunately, in most mills employing this condensing arrangement, the primary condensate does not go to the turpentine decanter. In these mills, the primary condenser is controlled to maintain the turpentine in the vapor phase for condensation in the secondary condenser. It should be possible to reduce the turpentine sulfur content by increasing cooling-water flow to partially condense turpentine in the primary condensate and decanting the combined primary and secondary condensate.

In recent months, turpentine recovery from the Union Camp mill at Prattville, AL, has improved while the sulfur content has decreased significantly. **Figure 8** shows data from 1989–93. Combining the primary and secondary condensates on one of the continuous digesters has increased the decantation temperature up to about 82°C (180°F). Laboratory studies have shown that increased temperatures break down turpentine emulsions. The results at UCC–Prattville confirm the laboratory work. With the increase in decantation temperatures, turpentine emulsions in the digester decanter have decreased dramatically. During the same period of time, the turpentine sulfur content has decreased. It is also possible that the higher de-

IV. Sulfur species in turpentine from Mills A and B, %

	Mill A			*Mill B*
	Turpentine to pressure	*Turpentine at pressure*	*Overall*	*Overall*
Methyl mercaptan	0	16	13	3
Low-boiling sulfur species [a]	80	75	70	81
High-boiling sulfur species [b]	20	9	17	16
Total	100	100	100	100

[a] Organosulfur compounds that boil below the boiling temperature of α-pinene
[b] Organosulfur compounds that boil at or above the boiling temperature of ß-pinene

V. Sulfur in turpentine—cold-blow digesters

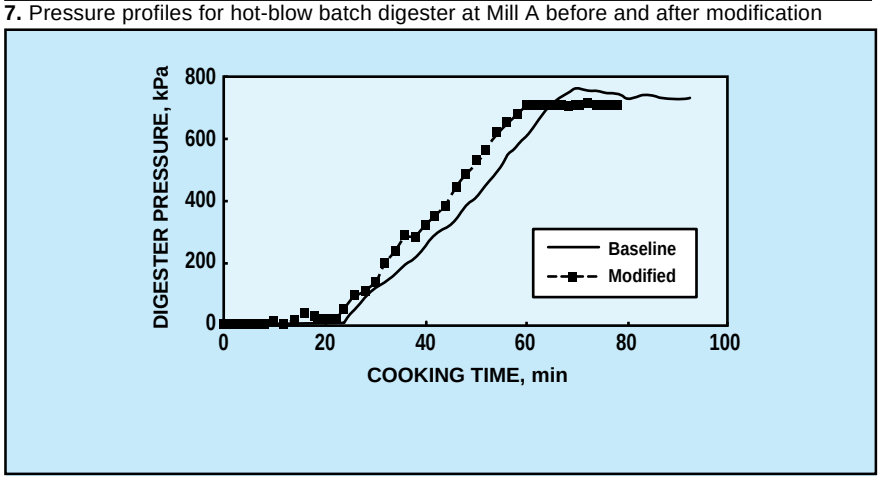
Mill	*Sulfur in CST, ppm*	*Turpentine yield, L/metric ton*	*Chip-bin presteam*	*Liquor carryover*
Bleached kraft				
Mill F (continuous, dual vessel)	34,436	0.96	Little	High
Mill G (continuous, single vessel)	29,420	1.88	None	Moderate
Mill H (continuous, single vessel)*	15,104	3.13	None	Low
Mill I (continuous, single vessel)*	10,149	3.13	Moderate	Low
Mill J (batch)	8,100	0.96	None	High
Unbleached kraft				
UCC–Prattville	6,979	2.59	None	Low
UCC–Savannah	4,068	1.84	Moderate	Low
Mill K	3,751	2.17	None	Low
Mill L	1,900	1.59	None	Low

* Data limited to 1990–91, since the UCC–Jacksonville facility no longer receives turpentine from these mills.

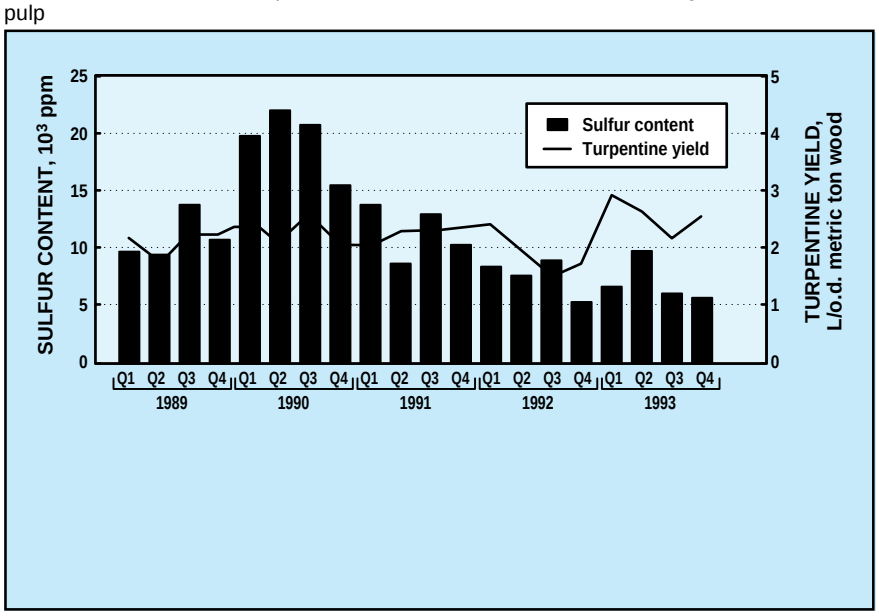
VI. Sulfur in turpentine for cold-blow digesters with two-stage turpentine condensing

	Primary-condensate turpentine	*Secondary-condensate turpentine*
Unbleached kraft (UCC–Savannah)		
Total S, ppm	1,200	5,000
Low boilers, %	84	94
High boilers, %	16	6
Total	100	100
Bleached kraft (Mill G)		
Total S, ppm	2,500	18,000
Low boilers, %	71	95
High boilers, %	29	5
Total	100	100

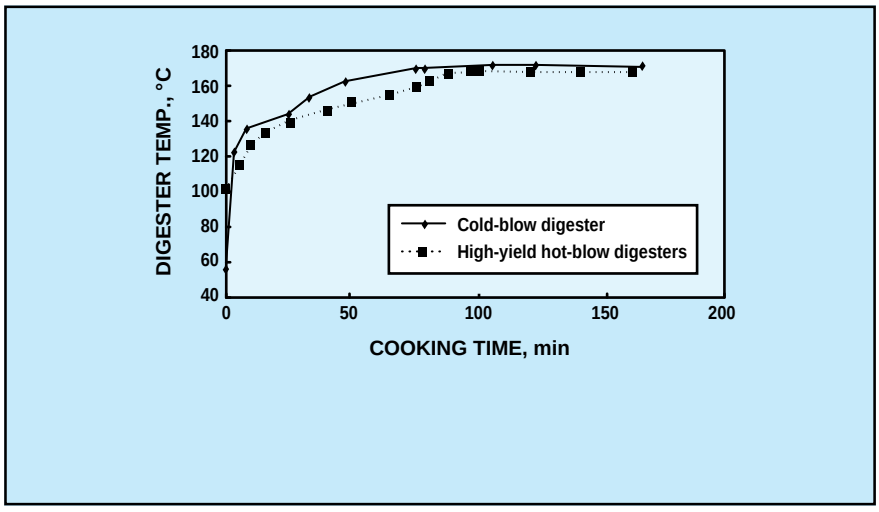
7. Pressure profiles for hot-blow batch digester at Mill A before and after modification



8. Sulfur content of kraft turpentine from UCC–Prattville continuous digester, unbleached pulp



9. Temperature profiles for hot-blow and cold-blow batch digesters



canter temperatures are driving off some of the reduced-sulfur compounds in the secondary-condensate turpentine.

With an increase in the condensate flow to the decanter, there is a greater opportunity for turpentine loss in the decanter underflow. This appears to be largely offset by better decanting efficiency, the result of reduced water viscosity because of the increased temperature of the combined condensate. Trials are being conducted on a low-cost compact decanter that would permit separate decantation of the primary and secondary condensate.

A troubling trend for turpentine processors is an apparent shift in the nature of the reduced-sulfur compounds present in turpentine to species that are more difficult to remove during fractionation. TRS compounds are primarily removed by steam stripping. Compounds with boiling points near the boiling points of the desirable terpenes are the most difficult to remove. **Table VII** shows that there are significant differences in the organic sulfur species present in turpentine from different types of continuous digesters. (Note that the total sulfur contents in turpentine from the mills in Table VII do not match those in Table V, since the data in Table VII represent a limited study rather than annual composite data.)

Cold-blow batch digesters

In recent years, several cold-blow batch pulping processes have been developed and installed *(3, 4)*. The heating profile to cooking temperature is comparable with the desired profile for the hot-blow batch processes, as seen in **Fig. 9**.

In contrast to the hot-blow batch processes, there is no vapor space at the top of the cold-blow batch digester when the turpentine begins to evolve before the cook begins. Instead, the turpentine-bearing liquors are vented to liquor accumulators during the liquor charging, cooking, and in-digester washing stages of

VII. Turpentine composition from continuous digesters, %

	UCC Savannah	UCC Prattville	Mill G	Mill H	Mill I
Light ends	0.2	1.2	2.3	1.9	0.8
α-pinene	58.0	60.6	62.9	57.1	63.0
ß-pinene	21.0	21.0	16.3	27.3	22.6
Other desirables	20.9	17.3	18.5	13.8	13.6
Total	100.1	100.1	100.0	100.1	100.0
Sulfur, ppm	1,689	23,784	38,739	36,021	12,320
Low-boiling TRS [a]	100.0	93.6	73.1	98.8	99.1
High-boiling TRS [b]	0.0	6.4	26.9	1.2	0.9
Total	**100.0**	**100.0**	**100.0**	**100.0**	**100.0**
Extended delignification	No	No	Yes	. . .	No

[a] Low-boiling TRS compounds are those boiling below the boiling temperature of α-pinene
[b] High-boiling TRS compounds are those boiling at or above the boiling temperature of ß-pinene

the cooking cycle. Turpentine evaporating from these accumulators is then vented to the turpentine-collection system. A turpentine balance on one such system *(5)* showed the turpentine recovery potential—3.12 L (0.75 gal)/o.d. metric ton of pine wood—to be roughly equal to that of good recovery from a continuous digester. It is nearly impossible to effectively presteam the chips in a cold-blow system. Therefore, increasing the decanter temperature is the most likely means of reducing the turpentine sulfur content.

Condensate strippers

Many mills now operate condensate strippers to reduce the odor and BOD (biochemical oxygen demand) of the condensate streams. This permits more varied recycle opportunities for the condensate or a more innocuous stream that can be sent to the waste-treatment system. The condensate streams fed to the condensate stripper often include evaporator condensates, blow-heat condensates, and underflow from the turpentine decanter. Depending on the stripper design, a foul or red oil can be produced at the reflux condenser. Because this foul oil contains a substantial portion of terpenes, many mills recycle the foul oil to the turpentine decanter. This practice seriously degrades the quality of the turpentine by increasing its sulfur content. More importantly, the nature of the reduced-sulfur compounds changes dramatically.

Many of the reduced-sulfur compounds in foul oil have boiling points above those of α-pinene and ß-pinene. Odorous chemicals and resins produced from α-pinene and ß-pinene have stringent odor restrictions. The foul (red) oil odor is far more offensive than that of typical CST. The offensiveness of this odor seems to be out of proportion to the increased sulfur content of the foul oil. Therefore, foul oil should not be considered to be turpentine and should not under any circumstances be recycled to the turpentine decanter. The data in **Table VIII** illustrate this point.

The foul oil has a fuel value of about 41,900 kJ/kg (18,000 Btu/lb) and is often burned in the lime kiln.

Summary

The average sulfur content of crude sulfate turpentine (kraft) supplied to Union Camp's Jacksonville terpene and aromatics plant has increased from 4400 ppm sulfur in 1989 to 7000 ppm sulfur in 1993. The sulfur in the turpentine is in the form of organic reduced-sulfur compounds. This increase in turpentine sulfur content has been associated with process modifications implemented to comply with federal, state, and local environmental regulations. However, the increase in sulfur content is of serious concern to turpentine processors.

Comparison of operating conditions at a wide range of mills reveals several strategies for reducing sulfur content in turpentine.

- Mills producing pulp from hot-blow batch digesters should increase the maximum steaming rate to 190–230 kg/h/m^3 (12–15 lb/h/ft^3) of digester capacity. This should greatly reduce the quantity of turpentine present in the blow heat, creating a more innocuous blow-heat condensate that can be reused more widely.
- Mills should avoid sending turpentine-bearing blow-heat condensates to the turpentine decanter.

VIII. Composition of turpentine and foul oil from Mill I, %

	Turpentine from continuous digester	*Foul oil from condensate stripper*
Light ends	0.8	2.2
α-pinene	63.0	47.0
ß-pinene	22.6	22.0
Other desirables	13.6	28.8
Total	100.0	100.0
Sulfur, ppm	12,320	20,525
Low-boiling TRS [a]	99.1	29.8
High-boiling TRS [b]	0.9	70.2
Total	100.0	100.0

[a] Low-boiling TRS compounds are those boiling below the boiling temperature of α-pinene
[b] High-boiling TRS compounds are those boiling at or above the boiling temperature of ß-pinene

- Mills should never return condensate-stripper foul (red) oil to the turpentine decanter.
- Mills can reduce the offensive odor of foul oil by maintaining the rectification-condenser overhead temperature above 91°C (195°F).
- Mills with continuous digesters can reduce turpentine sulfur content by increasing the use of fresh steam in presteaming of the chip bin.
- Mills can combine primary and secondary condensate for decantation to reduce sulfur in turpentine.
- Mills can increase the operating temperatures of the turpentine decanter to reduce sulfur contents with little impact on turpentine yield.

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