Oxalic Acid Process Chemistry
ECF Bleaching - Generation

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Oxalic Acid: Introduction

Oxalic Acid (Oxa) impacts operations and products

- Ca:OxA scale formation impacts performance of bleaching equipment
- Influences reduced water usage practices
- Ca:OxA deposits have been noted on paper machine

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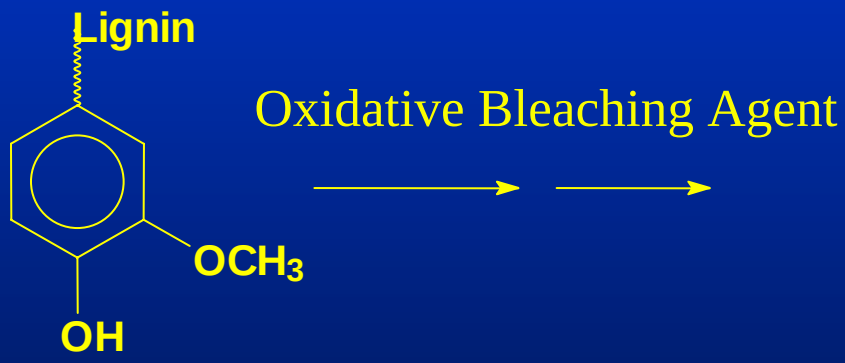
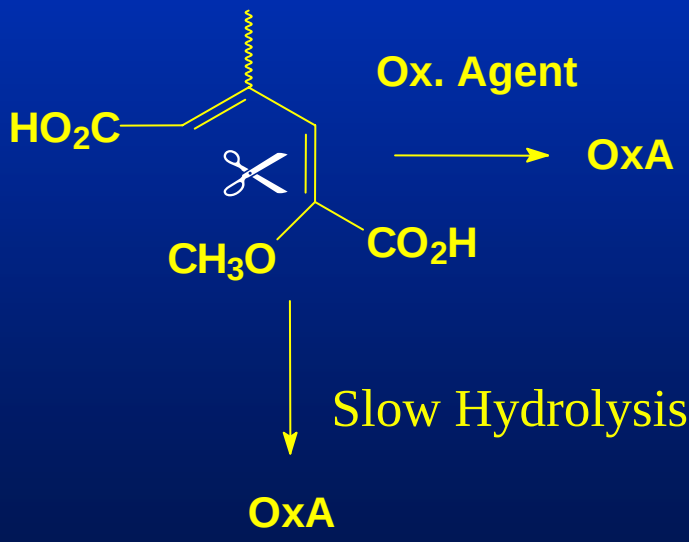
Oxalic Acid: Source

Wood:	Bark/ (kg/ton)	Wood (kg/ton)
HW	9 –15	0.1 – 0.3
SW	4 -10	0.1 – 0.4

Pulp





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OxA Accomplishments

- Developed CIE analysis method to analyze OxA in bleach effluents
- Demonstrated that [OxA] in bleach effluents can be increased upon heat treatment
    - OxA formation occurs under acidic and basic conditions
- Commercial HW D_0 effluents contained approx. 20 mg OxA/L
- Heat treatment of HW D_0 effluents increased [OxA] by 5 - 30%

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Oxalic Acid Generation Post-Do

- Do (kf:0.25) effluent from an OD SW kraft pulp

Reaction Time/h	**0**	**1**	**2**	**5**
Initial pH 2.6(70^{o}C)	**27**	**47**	**54**	**55**
Initial pH 11.5(70^{o}C)	**27**	**33**	**34**	**61**
Initial pH 2.6(90^{o}C)	**27**	**59**	**--**	**61**

[OxA] in effluents (mg/L)

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Oxalic Acid Research Goals

- Measure the amounts of oxalic acid in commercial SW D_0-stage effluents.
- Determine the effect of temperature on post bleaching formation of oxalic acid.
- Evaluate the effect of pulp kappa number on oxalic acid formation from a D_0-stage.

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Oxalic Acid Results: Commercial D_0 Effluents

Experimental Protocol

- Commercial samples acquired.
- Bleach effluent samples were heated to 70^{o}C for extended time period.
- Aliquots were removed and analyzed for oxalic acid content.

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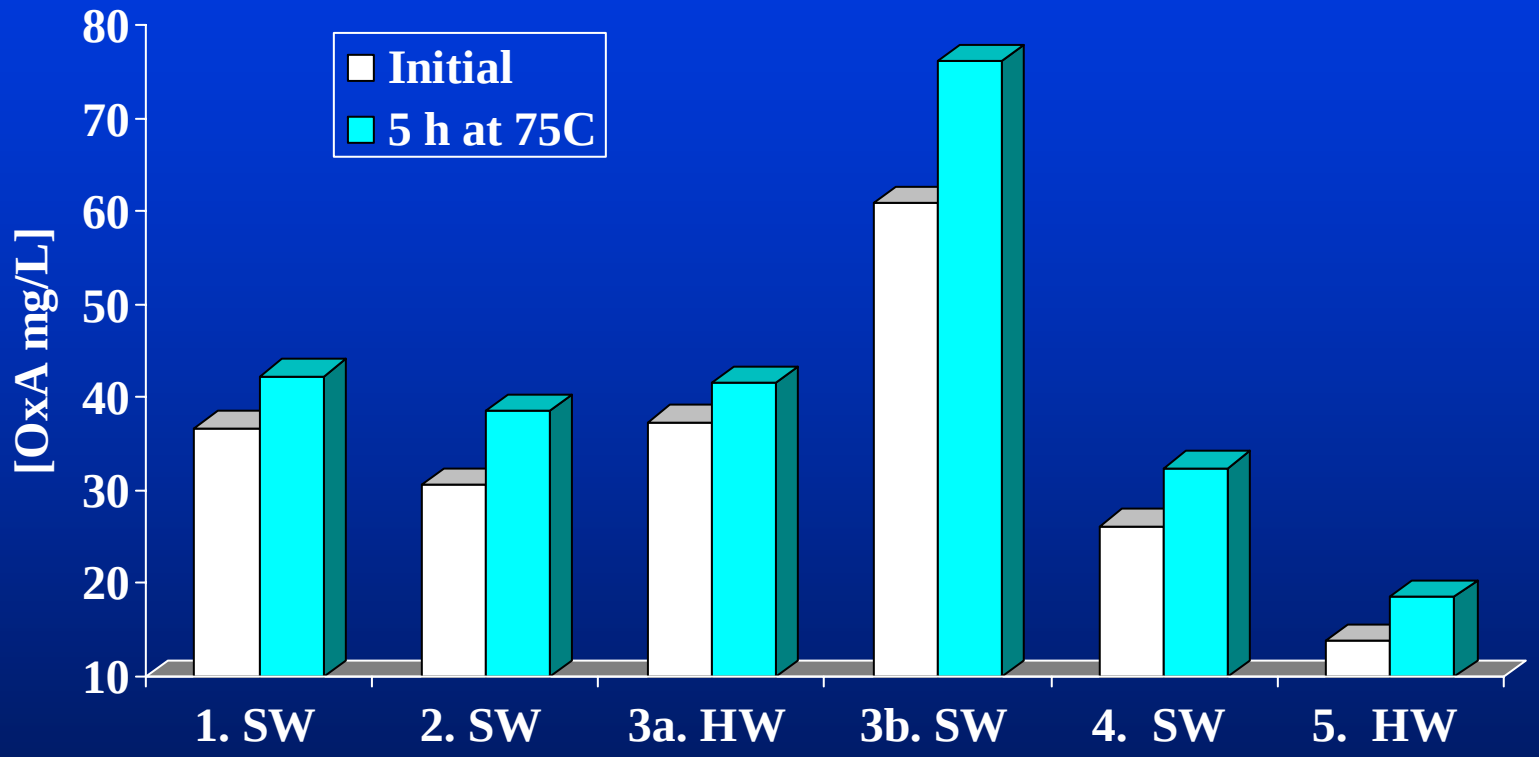
Oxalic Acid Results: Commercial D_o Effluents



Increase in OxA is in the range of 10 - 30%.

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Oxalic Acid Results: Laboratory Studies

Goal: Determine the influence of pulping and bleaching conditions on OxA formation.

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Oxalic Acid Results: Laboratory Studies

Experimental Protocol

Bleach series of SW kraft pulps with

0.1, 0.15, 0.20 kf ClO_2.

Pulps Employed

Conventional	Extended Modified Continuous
31.9, 25.1, 22.4	29.1, 23.2, 19.4, 17.0

-all from the same tree

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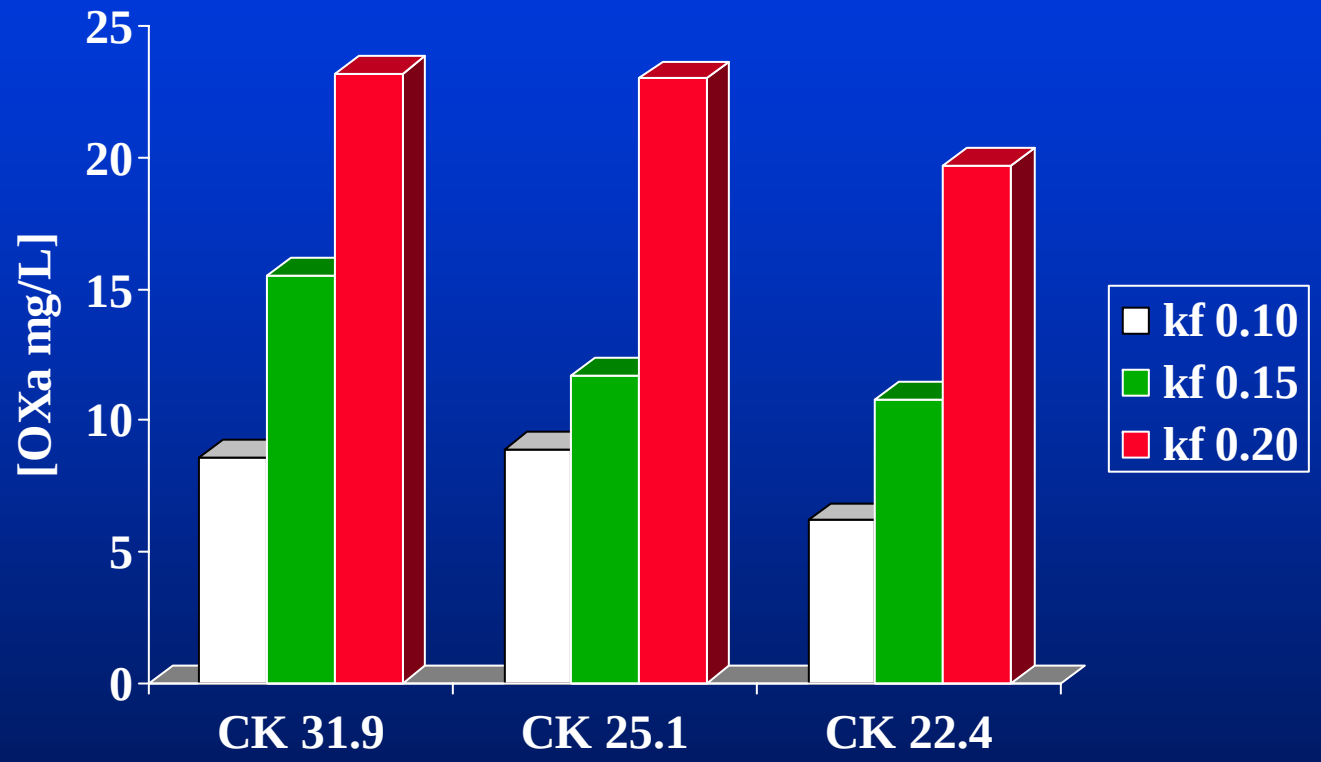
Oxalic Acid Results: [OxA] in D_0 Effluents from SW Conventional Kraft Pulp



2-fold increase in D-charge increases OxA by ca 2.8

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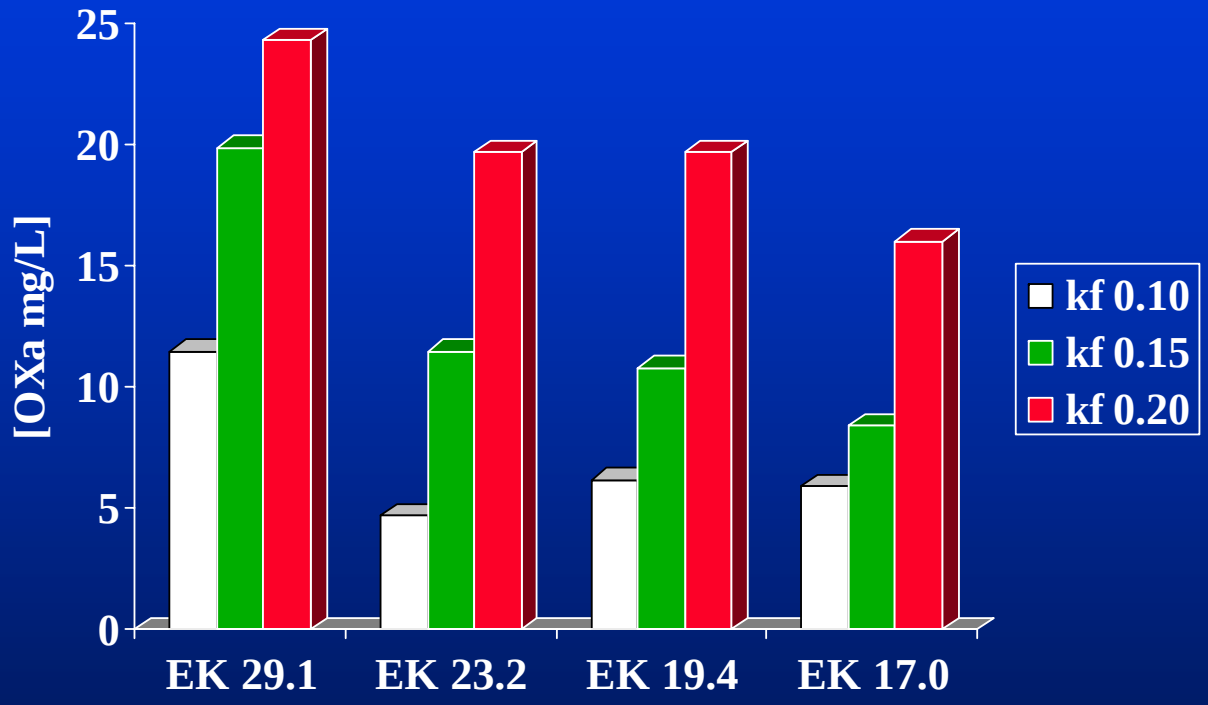
Oxalic Acid Results:
[OxA] in D_0 Effluents from Extended Modified SW Kraft Pulp



2-fold increase in D-charge increases OxA by ca 2.7

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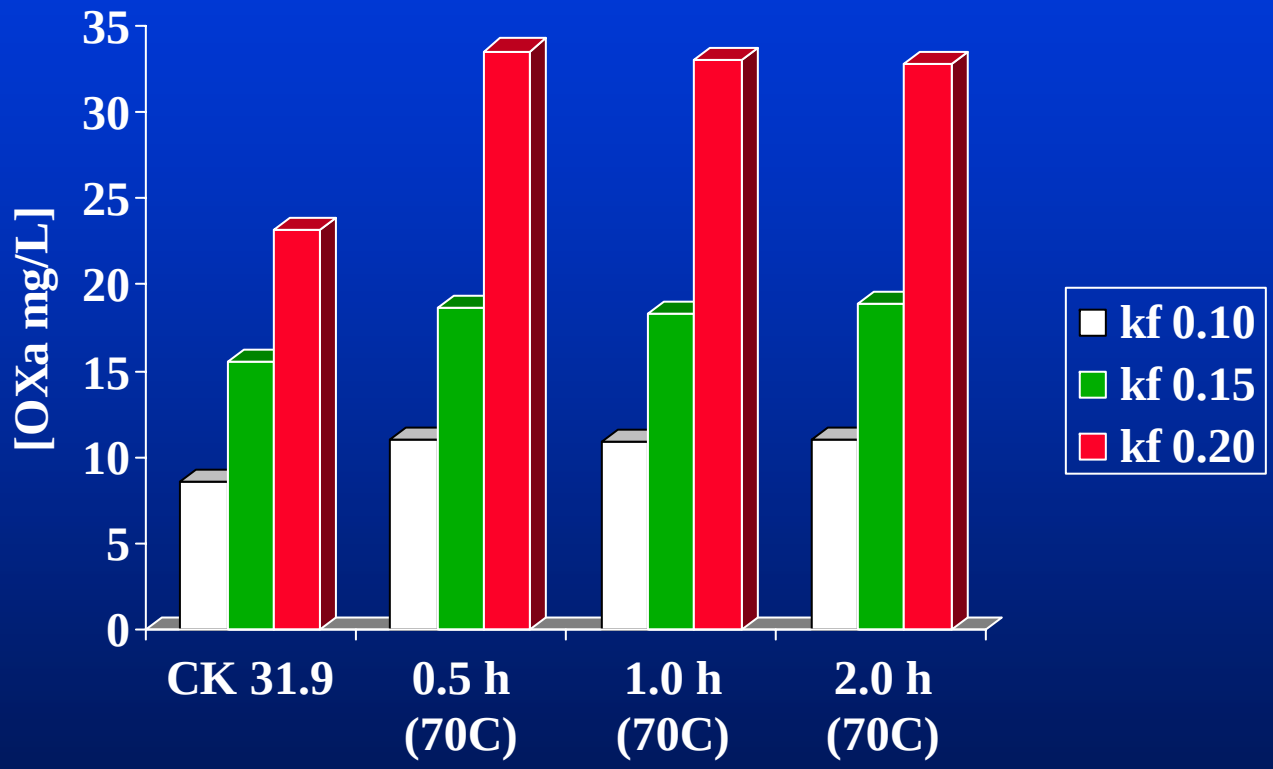
Oxalic Acid Results:
Heat Treatment of CK31.9 D_0 Effluent

Heat treatment of CK 31.9 D_o effluent at 70°C



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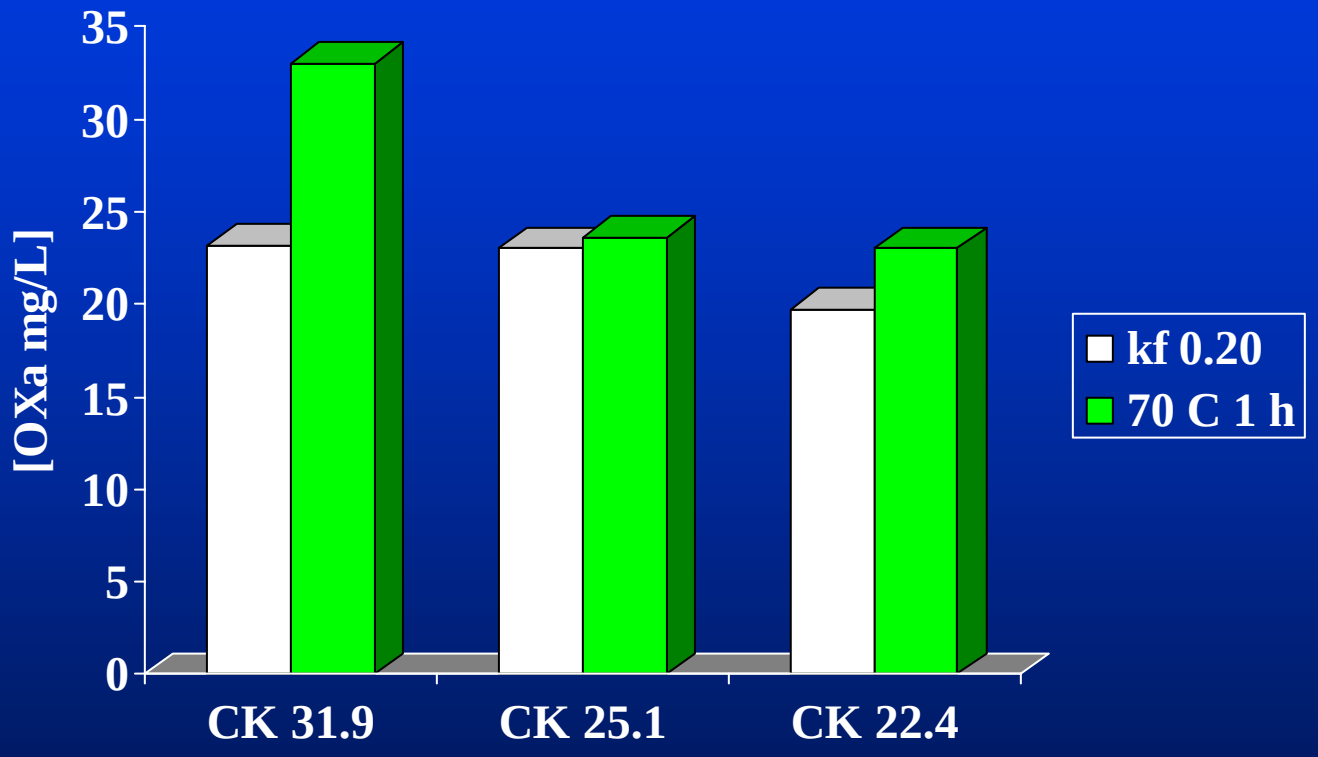
Oxalic Acid Results: Heat Treatment of CK D_0 Effluents

Results from heat treatment of D_o (0.20 kf) effluent from SW CK 31.9, 25.1, and 22.4.



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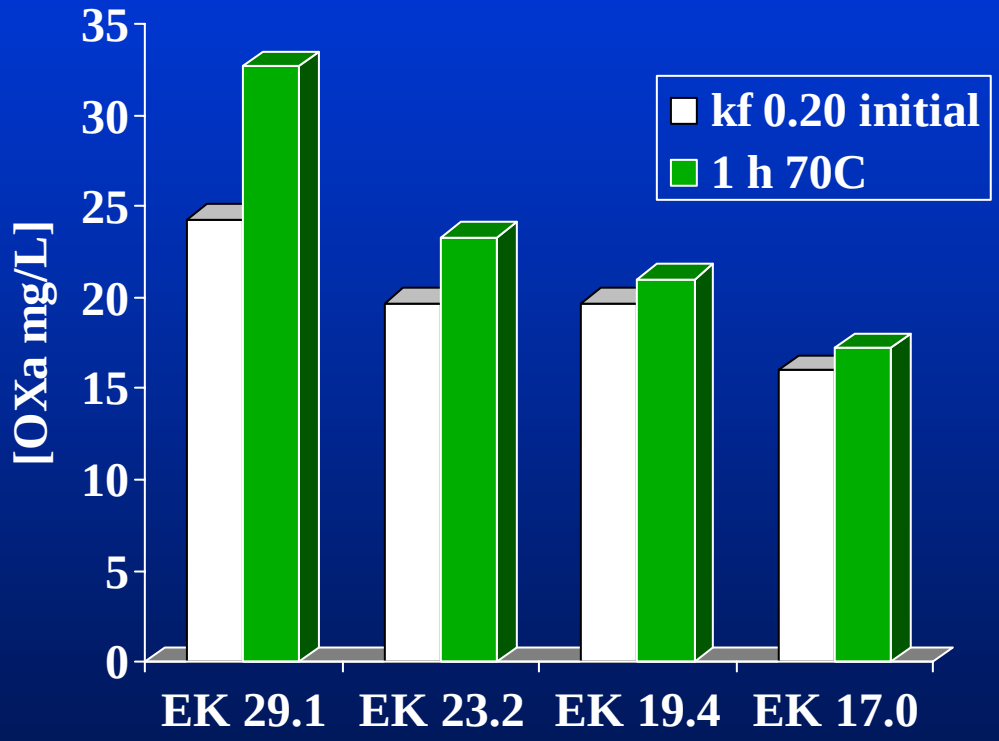
Oxalic Acid Results Results: Heat Treatment of EK D_0 Effluents

Results from heat treatment of D_o (0.20 kf) effluent from SW EK 29.1, 23.2, 19.4, and 17.0



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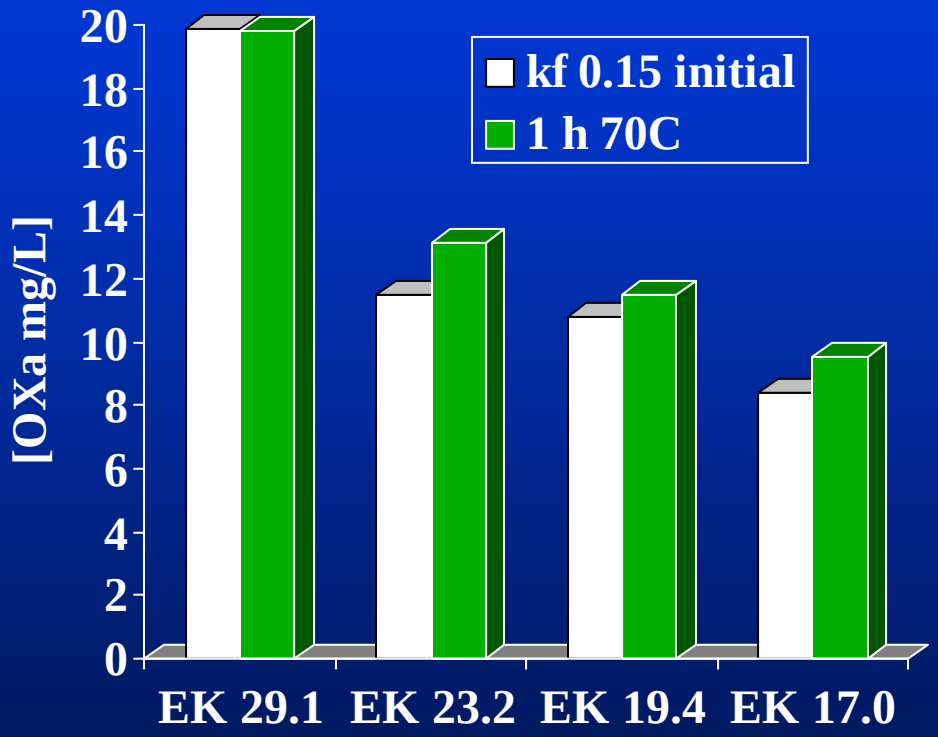
Oxalic Acid Results: Heat Treatment EK D_0 Effluents

Results from heat treating D_o (0.15 kf) effluent from SW EK 29.1, 23.2, 19.4, and 17.0.



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Oxalic Acid Conclusion

- Factors contributing to OxA
  - OxA generation is sensitive to D_0 kf
  - OxA generation is sensitive to starting pulp kappa #
- Heat generation of OxA
  - higher pulp kappa # and D_0 kf favors oxalic acid generation

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OxA Research: Value & Direction

- Any mill modeling of OxA-scale formation needs to take into account direct and indirect OxA formation chemistry
- Bench marked OxA in several member mills

- Generation of OxA through bleaching mill
- Affinity of OxA for fibers
- Control of OxA in bleached fiber line
  - develop new chemistry to control OxA

>>Yield process and product improvements

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