APPENDIX A

EXPERIMENTAL DESIGN AND PROCEDURES

Appendix A
Experimental Procedures

Apparatus

The oxygen delignification reactor and experimental techniques have been described previously by Guven et al. (1996) and Agarwal (1999, 1997). The experiments were performed in a 2-liter, medium consistency oxygen reactor that was equipped with a mixer designed to approximate the low level of mixing in an oxygen up-flow tower (Figure A1). The speed of the mixer was set at 23 RPM and was constant for all the experiments during the oxygen delignification reactions.

The reactor had previously been shown to simulate kappa number reductions seen in mill trials. The weight of the pulp used in these experiments was approximately 150-grams (dry basis). The oxygen delignification experiments were performed on well-washed pulp. No attempt was made to simulate the carryover of black liquor solids. All caustic was added prior to the first stage of the reaction.

Experimental Conditions

Experimental conditions are shown summarized in Figures A2 and A3. Two sets of experiments were performed corresponding to separating the effects of the pressurized reactor from the atmospheric tower. The first set of experiments simulated the conditions present in the 100 psig pressurized 1st stage reactor (Figure A2). The second set of experiments was performed to determine the extent of delignification taking place in the atmospheric tower (Figure A3).

15 Minute Reaction Vessel. To simulate the delignification in the 15 minute pressurized vessel, experiments were performed under the base conditions of 175 °F (79.4 °C), 10% consistency, 100 psig oxygen pressure, and 15 minute residence time and 1.5% caustic addition based upon pulp. The effect of consistency was determined by performing experiments at both 8 and 12% consistency under the base case. Similarly, the effect of temperature was determined by performing experiments at elevated temperatures of 190 °F (87.8°C) and 205 °F (96.1 °C) at 1.5% caustic addition rate. The effect of caustic addition rate was determined by performing experiments at 2.5%, 3.5%

and 4.5% NaOH caustic addition at 175 °F (79.4 °C). Nine (9) experiments were performed to simulate the reaction taking place in the 15 minute pressurized tower.

Two Stage Delignification. A second series of experiments were performed to infer the effects of leaching lignin from the pulp in the atmospheric tower. This was done by simulating exposure of the pulp to oxygen and caustic in both the 15 minute reactor and the atmospheric tower (Figure 6) and then by comparing the results to those obtained from exposure only in the 15 minute tower. The experimental procedure followed in this series of experiments was to expose the pulp to 15 minutes in the high pressure tower at 100 psig and then quickly releasing the pressure to atmospheric pressure to simulate the blow tank. The pulp was then pressurizing to 20 psig with oxygen and gradually the pressure was reduced according to the schedule shown in Table A1.

Table A1.
Pressure Reduction to Simulate the Atmospheric Tower

Time (Minutes)	Pressure (Psig)
0 s	20
12	16
24	12
36	8
48	4
60	0

The pressure reduction schedule was designed to simulate the hydrostatic pressure in the atmospheric tower and the gradual reduction as the pulp proceeded up the tower. In these experiments, experiments were performed at both 10 and 12% consistency and both 175 and 190 °F. No attempt was made to vary the temperature between the two towers. The effect of changing caustic was simulated by performing experiments at both 2.5% and 3.5% caustic addition rates. A total of eight (8) experiments were performed to simulate the two stage process taking place at Jay, Maine.

Response Variables After Oxygen Delignification

Following exposure to oxygen, the pulp was sampled after each experiment for residual liquor and then thoroughly washed and stored. Following storage, handsheets were prepared and used for testing. The response variables that were measured after oxygen delignification were (1) kappa numbers, (2) intrinsic and 0.5% TAPPI CED viscosities, (3) brightness, (4) residual alkali, (5) pH, and (6) the COD, and (7) Color contents of the squeezed liquor.

Physical Tests

The kappa number for the pulp samples was measured according to TAPPI standard method T-236. The intrinsic viscosity [η] was estimated according to ASTM standard D1795-62 which is similar to SCAN-C 15:62, while the TAPPI 0.5% CED viscosity was determined by following TAPPI standard method T-230. The equation of Evans and Wallis (1989) can be used to relate the degree of polymerization (DP) to the intrinsic viscosity [η] to the degree of polymerization of the carbohydrates.

$$DP_V^{0.9} = 1.65 * [\boldsymbol{h}, cc / gram] \qquad \text{(A1)}$$

ISO brightness was determined using a Technibrite Micro TB-1C brightness meter. Residual alkali was determined by titration following TAPPI Standard T-625. The COD measurements were determined by digesting the spent liquid with potassium dichromate using the method and apparatus purchased from the Hach Company. Consumption of potassium dichromate was determined by colorimetric methods as described by Gibbs (1979). Color was determined spectrophotometrically by following Hach Method DR 8025. The Hack method measures true color and follows Method 204B found in Standard Methods for the Examination of Water and Wastewater (1975).

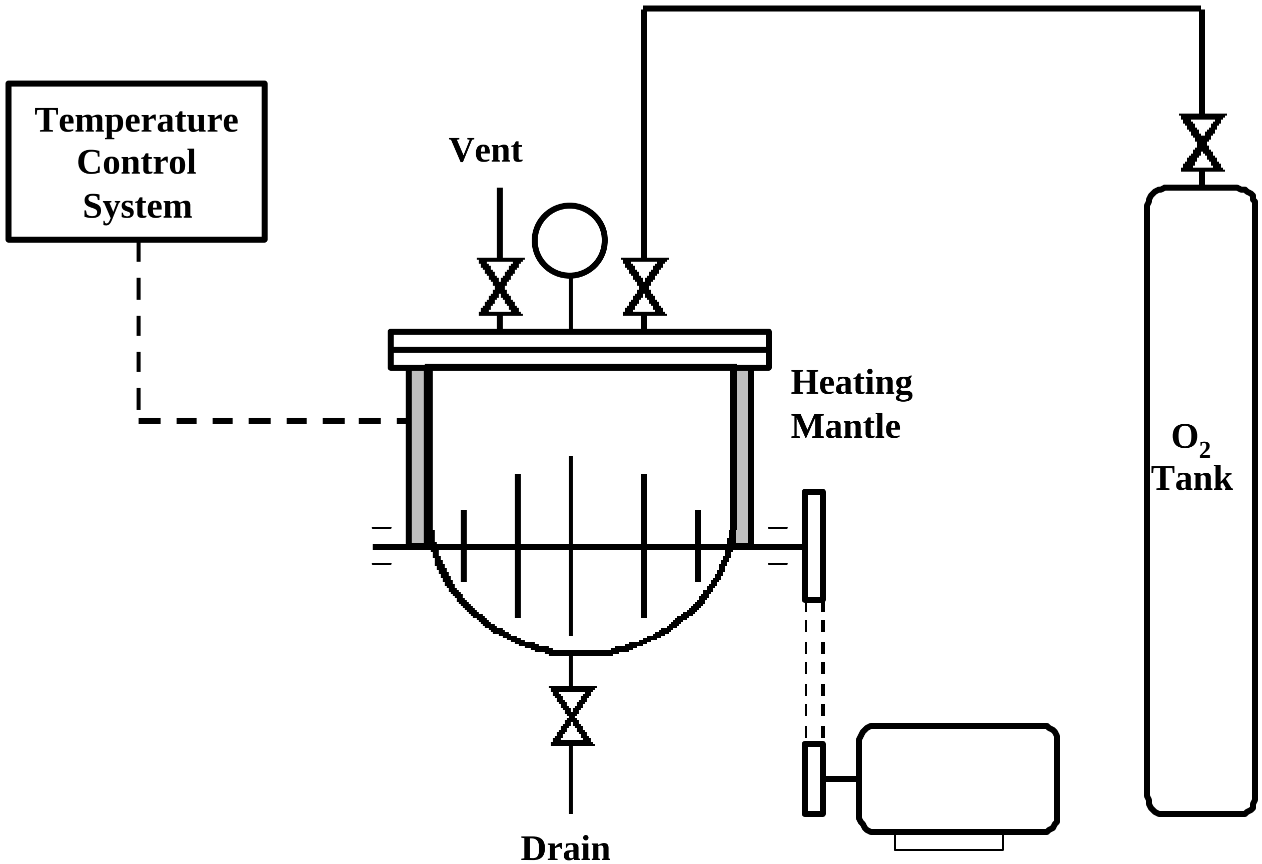

Figure A1
Schematic Diagram of UM Oxygen Delignification Reactor

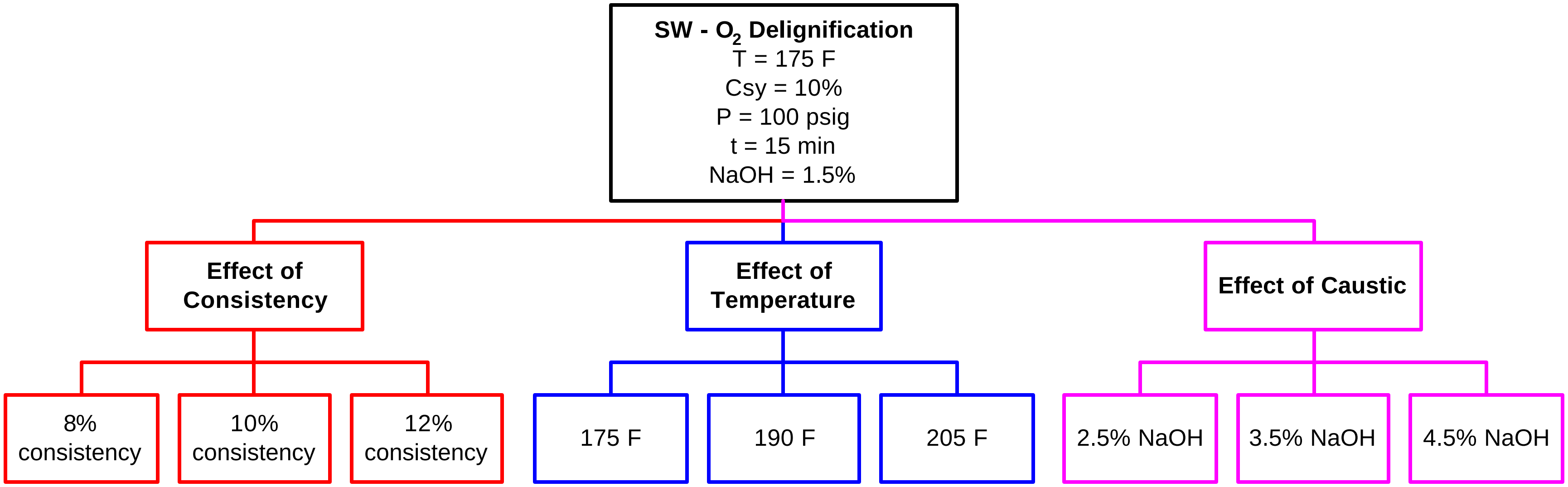

Figure A2.
Experimental Design to Simulate 1st Stage Reaction Vessel

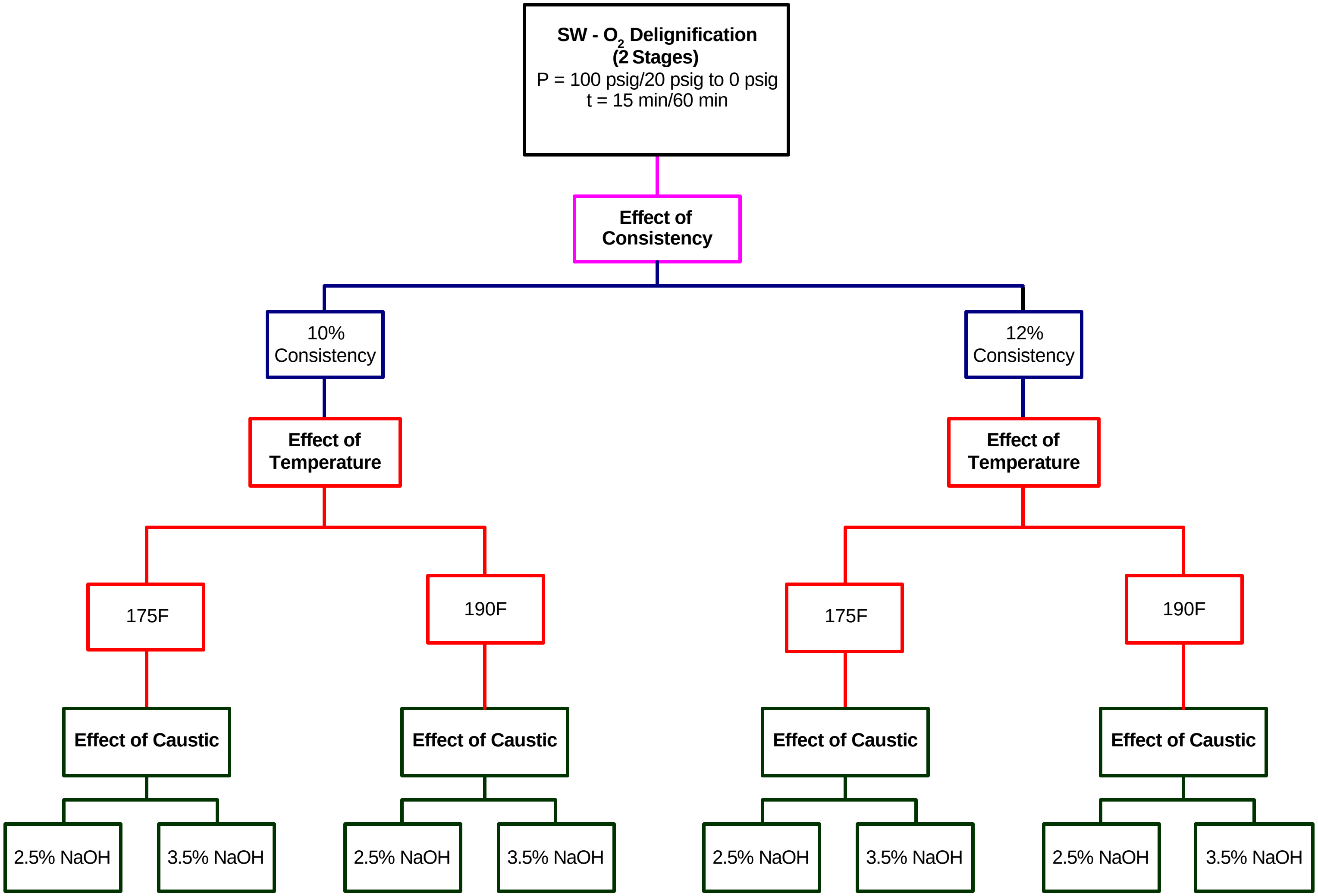

Figure A3
Experimental Design to Simulate Effect of the Atmospheric Tower