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## The Effect of Infra-Red Radiation on the Curing Rate of an Alkylketene Dimer Sizing Agent

Roland A. Johnson  
*Western Michigan University*

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THE EFFECT OF INFRA-RED RADIATION  
ON THE  
CURING RATE OF AN ALKYLKETENE DIMER  
SIZING AGENT

by  
Roland A. Johnson

A Thesis submitted to the  
Faculty of the Department of Paper Technology  
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of the  
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## ABSTRACT

The contents of this thesis examine the principles behind and the effects of the application of infra-red radiation on a sheet sized with an alkylketene dimer. Handsheets were formed by the Noble and Wood procedure from bleached softwood kraft pulp beaten to a Canadian standard freeness of 390. They were sized with the dimer to a 0.2% concentration on the weight of the oven dry fiber combined with a cationic retention agent to a 0.05% concentration. It was found that infra-red radiation improved the sizing effects of the dimer.

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## THEORY

### The Alkylketene Dimer

The alkylketene dimer is prepared from condensation of long chain fatty acids such as: palmitic, stearic, oleic, and myristic acid. The stearic acid derivative configuration is as follows:

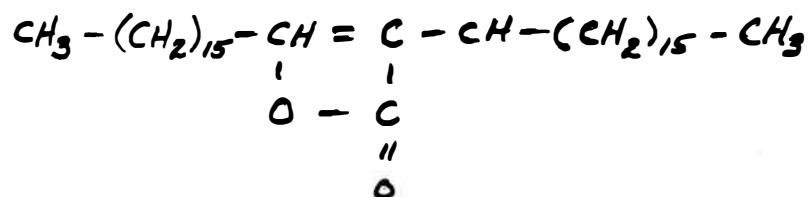


Figure 1

Under alkaline conditions, the lactone ring opens up and is free to react with any free hydroxyl or amine groups. The  $\beta$ -keto ester formed during this reaction is responsible for the sizing characteristics of the dimer.<sup>1</sup> Following is a sketch of the above ester as it might appear in paper.<sup>2</sup>

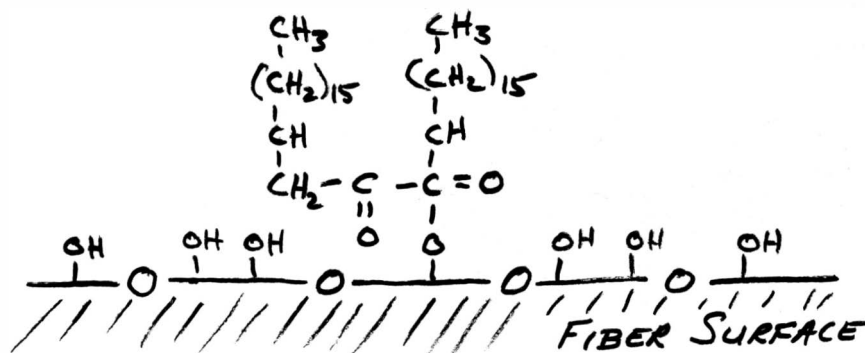


Figure 2

The alkylketene dimer can undergo side reactions which would alter its sizing efficiency. With water, it forms a

ketone. It is only after the water has been removed that the dimer begins to react with the cellulose, and then only 60% to 75% of that retained will form the ester. It is estimated that on most machines, only 35% of the dimer retained is actually consumed by hydrolysis and often this reaction occurs over a span of 1 to 3 days.<sup>1</sup>

Kincannon, Perekal'skiy, Antonovich, Krukova, and Kurylev suggest that maximum sizing efficiency may be achieved by increasing the steam pressure in the first dryer section (tempering).<sup>2,3</sup> Kincannon studied the rate of aquapel sizing from the standpoint of temperature dependence and discovered that the time required to reach a specific reading on the Hercules Sizing Testor was substantially reduced when the drying temperature was increased.<sup>2</sup> A plot of his data follows:

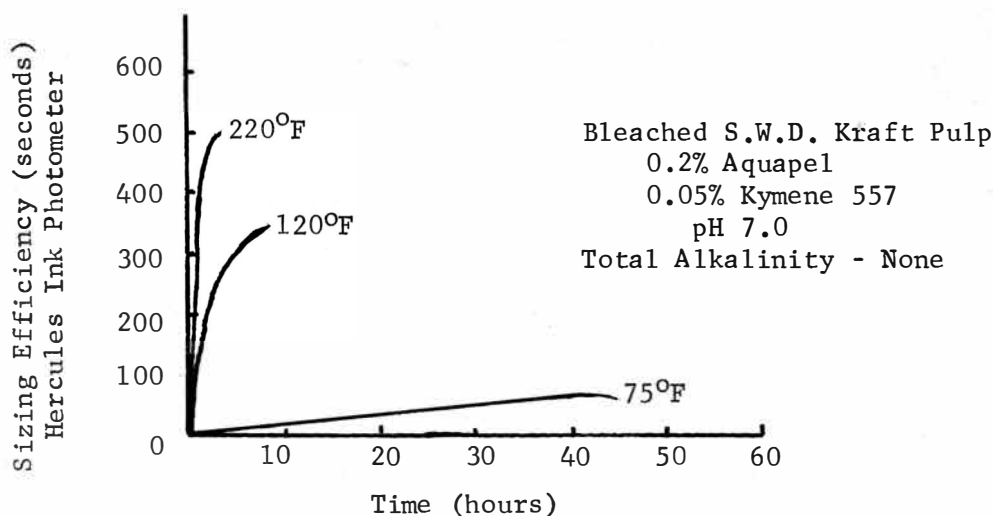


Figure 3

The sizing mechanism is also pH dependent. "Below a pH of 7.0, the rate of reaction is retarded and sometimes the degree of sizing is adversely affected. Above a pH of 7.0, the rate of reaction increases with increasing pH."<sup>1</sup>

### Infra-Red Radiation

Infra-red radiation is often referred to as thermal radiation and obeys the same laws as does light.<sup>4</sup> All substances at temperatures above absolute zero emit radiation of this type. The energy radiated from a substance is measured against that emitted by a black body which emits uniformly at all wave lengths.<sup>5,6</sup> This ratio is called the emittance ratio and when plotted against the wavelength, Figure 4 results.

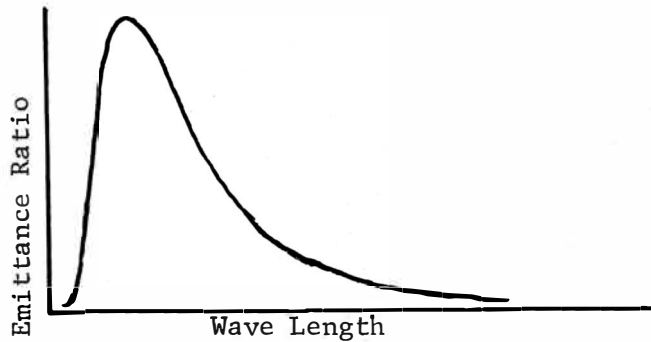


Figure 4

Black-body radiation is described by the Stefan-Boltzmann law which states that the energy emitted by a surface is proportional to the temperature of the surface, or

$$U = A T^4$$

where A is a factor.<sup>5,6</sup>

The distribution of this energy in the spectrum is given by Planck's law.<sup>5</sup>

Although all substances emit radiation of this type, not all of them absorb energy at every wavelength. It would seem necessary to investigate the absorption characteristics of the system being used for this thesis.

Figure 5 is a diagram of the absorption characteristics of a water film at various thicknesses.<sup>5,7,8,9</sup> One can see that as the film gets thinner, it becomes increasingly translucent to thermal radiation.

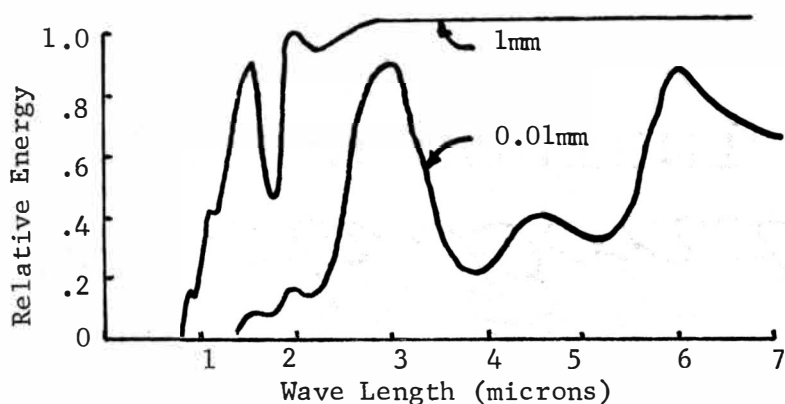


Figure 5

Figure 6 is a diagram of the absorption characteristics of the cellulose fiber.<sup>7</sup> The fiber absorbs in the same regions and thus, a system composed of the two would behave similarly to radiation of the same wavelength.

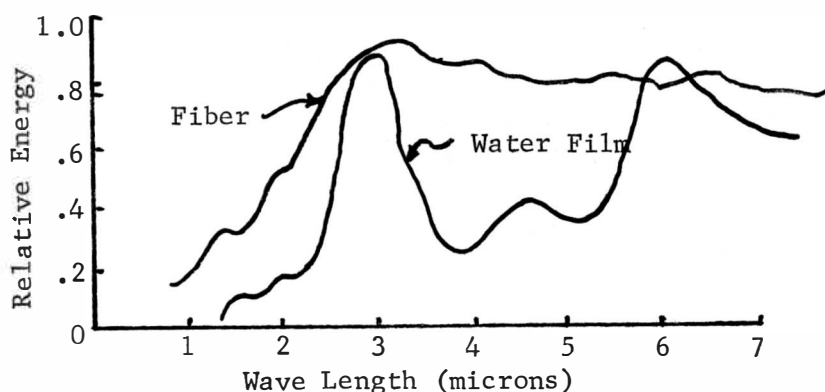


Figure 6



## DEVELOPMENT OF THE PROBLEM

It is the characteristic problem of alkylketene dimer sized paper that it has not reached its maximum sizing efficiency when tested off of the reel. In fact, the sizing reaction is not completed until 1 to 3 days after manufacture, and sometimes longer, resulting in quality control problems.

It has been stated in the discussion under theory, that the dimer will react with the free hydroxyls of water as well as with those of cellulose. It would seem reasonable, then, that by removing the water as soon as possible, the reaction with water is limited and the reaction with the cellulose is encouraged. It is also shown that as the drying temperature is increased, the sizing efficiency of the dimer is increased.

Since infra-red radiation delivers a larger amount of energy per unit of time than normal conventional paper drying methods, the water suspended in the sheet can be evaporated faster. Thus, it is assumed that infra-red radiation can be used to accelerate the sizing reaction of the dimer system. The following procedures and data are an effort to substantiate this assumption.

## EXPERIMENTAL PROCEDURES

### Handsheet Formation

A bleached softwood kraft pulp was beaten at a consistency of 1.50% to a Canadian Standard Freeness of 390 in a laboratory Valley beater. A portion of this slurry (50 grams oven dry fiber) was placed in the handsheet proportioner. A cationic retention agent, Kymene 557, was added at a rate of 0.05% on the weight of oven dry fiber.<sup>1,2</sup> The liquid level was then adjusted to yield a Noble and Wood handsheet of 2.50 grams. Aquapel, at the rate of 0.2% on the weight of the oven dry fiber, was then added in the sheet mold before further dilution. The suspension was diluted and then drained. The sheets were formed at a pH of 6.8 and dried according to the scheme given in the next section.

### Drying Scheme

Four series of handsheets, consisting of fifteen handsheets each, were prepared using standard Noble and Wood apparatus. After wet pressing, the first series was allowed to air dry at 72° F and a relative humidity of 50%. The second and third series were dried to a moisture content of 3.2% on the drum drier at temperatures of 180° F and 232° F,<sup>2</sup> respectively. The fourth series was dried using infra-red radiation,<sup>10</sup> also to a moisture content of 3.2%.

In drying series four, the sheets were placed five inches

beneath the infra-red source for twenty-five seconds. Once dried, each series was conditioned at 72° F and 50% relative humidity and then tested according to the following procedure.

#### Sizing Efficiency Measurement

Each handsheet was divided into four (4) sections and the sizing efficiency was determined on ten (10) samples from each drying series on the Hercules Ink Photometer which measures the time, in seconds, for the standard testing ink to penetrate the sheet and reduce the reflectance to 80%.<sup>11</sup> Each series was evaluated accordingly:

<u>Drying Series</u>	<u>Testing Schedule</u>
Air dried	12, 20, 36, 42, 60 hours
180° F	2, 4, 6, 8, 24 hours
232° F	1, 4, 6, 8, 25 hours
Infra-red	2, 4, 6, 8, 26 hours

Table 1

## DATA

<u>Drying Series</u>	<u>Testing Point (Hours)</u>	<u>Average Sizing Efficiency (Seconds)</u>
Air Dried	12	2.3
	20	3.7
	36	1.4
	42	1.8
	60	1.6
180° F	2	5.0
	4	7.1
	6	8.5
	8	10.2
	24	15.5
232° F	1	6.4
	4	12.7
	6	17.7
	8	19.6
	25	22.0
Infra-red	2	12.1
	4	16.9
	6	19.6
	8	19.5
	26	26.1

Table 2

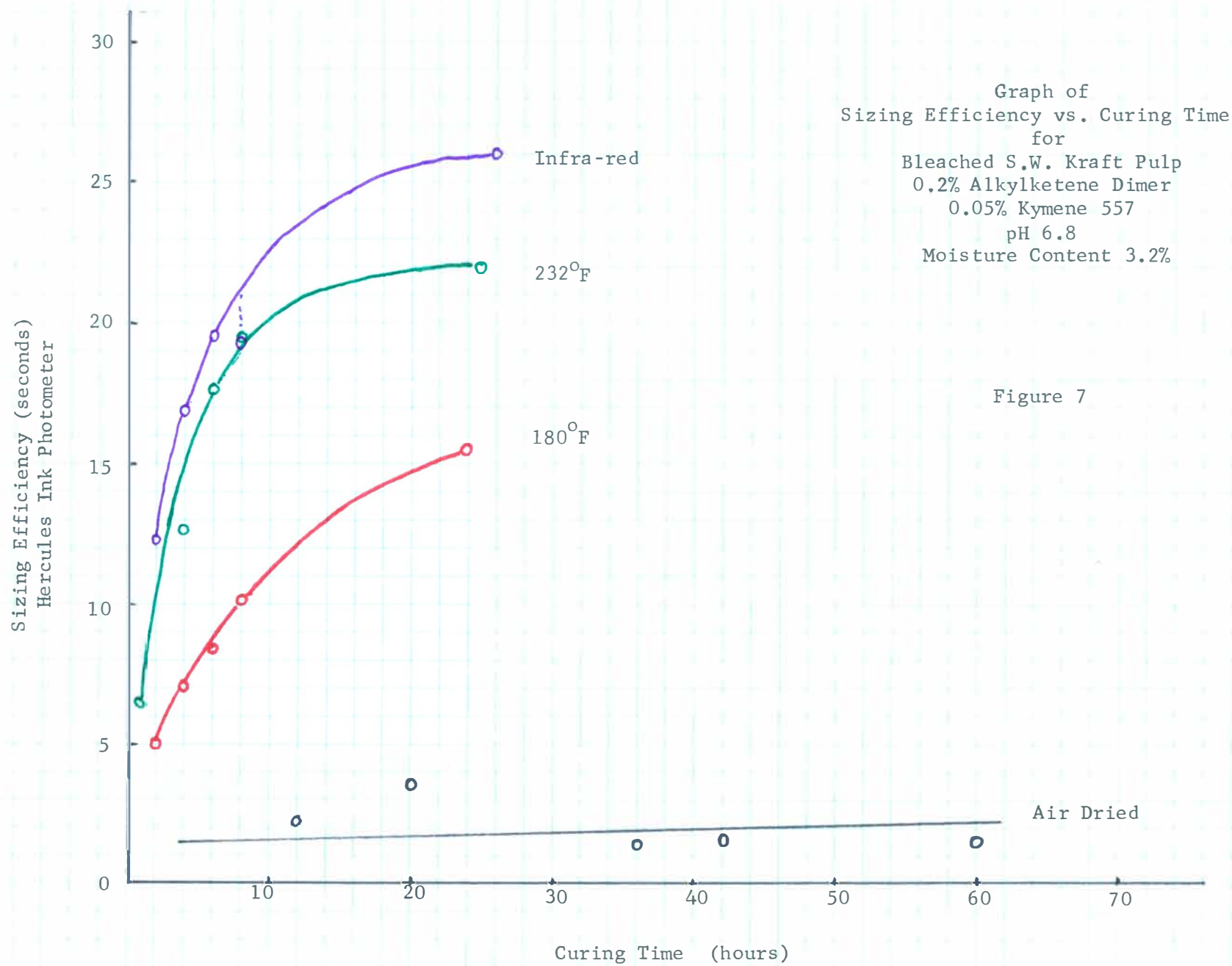


Figure 7

## DISCUSSION OF RESULTS

It must be noted that the infra-red source was a 9-inch, 220 volt, electrically powered laboratory element and that the dominant wavelength of radiation was not determined since a cost comparison is not attempted here.

To make the task of forming the handsheets easier, the pH of the suspension was not adjusted to achieve the maximum sizing effect. Instead, iron free tap water at a pH of 6.8 was used. Since the dimer reaction occurs best above a pH of 7.0, this data cannot be quantitatively compared to other work. However, a quantitative evaluation can be made.

In the work done by Kincannon,<sup>2</sup> it is apparent that by increasing the drying temperature, the sizing efficiency increases. The data illustrated in Figure 7 also reveals this and indicates that infra-red radiation does favorably affect the sizing efficiency of the alkylketene dimer. This means that for any given length of time, a sheet dried with infra-red yields a higher sizing value than one sized by conventional means.

## CONCLUSIONS

The general conclusion drawn from this thesis is that infra-red radiation, when used during the drying process of a sheet sized with an alkylketene dimer, improves the sizing efficiency. It is suggested that it does so by removing the water rapidly, thus leaving the alkylketene dimer free to react with the free hydroxyls of the cellulose.

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