EXPERIMENTAL INVESTIGATION OF SMOLDERING COMBUSTION
OF CELLULOSIC MATERIALS

by

MARCOS G. ORTIZ MOLINA

"Ingeniero Mecanico"
Universidad Central de Venezuela
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Certified by

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ABSTRACT

Experiments were conducted to examine the effects of environment and fuel characteristics on the smoldering behavior of cylindrical samples of cotton. Environments of Nitrogen-Oxygen, Helium-Oxygen and Argon-Oxygen, with different oxygen partial pressures and mole fractions were employed. Samples of cotton thread with densities varying from 0.06 gr/cm$^3$ to 0.8 gr/cm$^3$, and diameters from 0.04 cms to 3.5 cms were tested.

Depending on the environment and the fuel characteristics three regimes of smoldering were observed: steady smoldering, transition to flame, and transition to extinguishment. For the case of steady smoldering theoretical calculations were performed, using a recently developed theoretical model of smoldering. These calculations seem to agree well with the experimental results. In the non-steady cases it was found that helium has a strong effect in the extinguishment and flaming limits found in N$_2$-O$_2$ environments. In He-O$_2$ environments the extinguishment limit conditions are found at much higher oxygen concentrations than in N$_2$-O$_2$ environments. Experiments in He-N$_2$-O$_2$ environments indicate that helium has an effect on the extinguishment limit. Then the possibility of using helium as an extinguishant for smoldering fires is examined and it is compared to other existing procedures of fire extinguishment.

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CHAPTER I

INTRODUCTION

Smoldering is a flameless form of combustion characterized by thermal degradation and charring of the virgin material, usually accompanied by evolution of heat, smoke, and emission of visible glow. The purpose of this thesis is to study the mechanism of smoldering combustion of cellulosic materials, and from the knowledge of the mechanism to propose more effective means of prevention and suppression of smoldering fires. Cellulosic materials have been selected for this study because: they are abundant and can be found in homogeneous forms which are able to sustain smoldering, and because their chemical properties (pyrolysis) have been investigated [1.1], so that theoretical analysis is possible.

Smoldering combustion plays an important role in the development of unwanted fires. Statistics show [1.2] that 75% of all dwelling fires can be expected to be smoldering in nature during their initial stage. Also, 9 out of 31 reported [1.3] aircraft cabin fires started being smoldering fires. It has also been reported [1.3, 1.4] that conventional fire extinguishant procedures are sometimes impractical, slow or ineffective in smoldering fires. Therefore the motivation for this study.

Several aspects of smoldering combustion have been studied before: the smoldering combustion of industrial dust heaps was
recognized as a factor in industrial fire hazards and motivated
the work of L. Cohen and N.W. Luft [1.5] in 1955, and K.N. Palmer
[1.6] in 1967. They studied the smoldering combustion of several
industrial dusts including metallic dusts (coal, cocoa, sawdust,
pyrites, cork, magnesium, etc.). They examined the relationship
between the smoldering speed, the particle size and the depth of
in 1965 studied the smoldering of a cigarette under alternating
natural and forced convection fields (as in smoking). They were
interested in the temperatures in the reaction zone, and in the
products of reaction. In 1967 Kimbara, et al [1.9] studied the
smoldering of cylindrical rods of paper, incense sticks and strips
of cardboard. They examine the effect of ambient temperature and
radii of the sample. They developed an approximate model of
smoldering which assumes some "ignition temperature", and that
the reaction is governed by the oxygen availability. Their model
seems to give good agreement with their experiments. More recently
in 1975 A. Moussa [1.10] studied smoldering of cylindrical samples
of cellulosic materials under different environmental conditions
and developed a theoretical model which includes the pyrolysis analysis
and the kinetics of the oxidation reaction. This theoretical model
is able to describe steady smoldering situations from the knowledge
of fuel and environment characteristics. Even though the model
does not describe any transient behavior, it determines extinguishment
conditions. This model has been selected to obtain theoretically the experimental results of this thesis.

In this thesis experiments were conducted mostly in cylindrical samples of cotton, to examine the effect of fuel and environment properties on the smolder behavior. A few experiments were conducted to determine the effect of initiation on smoldering, and to examine the pressurization with inert gas as a method to induce extinguishment. Chapter II is concerned with the description of the model and the assumptions employed to adjust the model to the experimental parameters. It describes the steps of a calculation, and the theoretical predictions are presented in figures together with the experimental results for comparison. Chapter III describes the experimental apparatus and the experimental procedure followed in the tests. It also presents the discussion of the experimental results which are usually measurements of smoldering, speed and temperature, and visual observations of the smoldering behavior under different environments. The effects of density and diameter of the sample are examined, as well as the effect of initiation heating rate and the initiation heat source. Of all these effects special attention is given to the effect of helium, which seems to favor extinguishment and disfavor transition to flaming. From this result it is proposed to examine the application of helium to extinguish smoldering fires. In Chapter IV
this possibility is studied and is compared with existent methods of fire extinguishment to find the advantages and disadvantages of using helium instead of other procedures.
CHAPTER I

REFERENCES


CHAPTER II
THEORETICAL MODEL

The purpose of this chapter is to introduce a general description of the theoretical model selected. It will also contain the equations and assumptions as they were applied to idealized situations in order to get some predicted smoldering behavior.

2.1 MODEL DESCRIPTION

This model [2.1] divides the smoldering combustion problem in two zones (Fig. 2.1), separated by an interface which must meet some required conditions to match the two zones.

Essentially the model states that there is a pyrolysis zone where the material decomposes due to heating, yielding char and volatiles as products, and there is a char-oxidation zone where the char produced in the pyrolysis process reacts with the oxygen of the environment generating heat. Part of this heat is feedback into the pyrolysis zone to sustain the pyrolysis process, and part goes to the environment as heat losses.

The analysis of each of these two theoretical zones is described in the following two sections.

2.1.1 Pyrolysis Zone

In order to describe the behavior of the pyrolysis zone it is assumed the occurrence of two simultaneous reactions [2.2]. A high activation energy reaction which gives volatiles as products and a low activation energy reaction that produces char. With this assumption, the steady propagation of a pyrolysis zone at the
at the speed \( V_s \) through a semi-infinite slab of material under the effect of an external heat flux imposed at the surface is studied.

Figure 2.2 is a physical description of the problem in which the pyrolysis zone is considered stationary and the material moves at the speed \( V_s \) into the zone. The mathematical formulation of this problem is:

Material continuity equations:

\[
V_s \frac{d\rho_u}{dx} = -\rho_u (K_c + K_g) \quad (2.1)
\]

\[
V_s \frac{d\rho_c}{dx} = \rho_u K_c \quad (2.2)
\]

\[
\frac{d(\rho V_g)}{dx} = \rho_u K_g \quad (2.3)
\]

Energy equation:

\[
\rho_\infty c_v V_s \frac{dT}{dx} = \lambda \frac{d^2T}{dx^2} + \rho_u (H_K c_c + H_g K_g) \quad (2.4)
\]

Rate equations:

\[
K_c = f_c \exp\left(-\frac{E_c}{RT}\right) \quad (2.5)
\]

\[
K_g = f_g \exp\left(-\frac{E_g}{RT}\right) \quad (2.6)
\]
With the boundary conditions:

\[ \text{at } x \to -\infty: \quad \rho_u \to \rho_\infty \quad (2.7) \]

\[ \rho_c \to 0, \quad (2.8) \]

\[ T \to T_\infty. \quad (2.9) \]

\[ \text{at } x = 0: \quad \frac{dT}{dx} = \frac{q_s}{\lambda}, \quad (2.10) \]

\[ \frac{\rho_u}{\rho_\infty} = \beta \ll 1 \quad (2.11) \]

where

\( V_s \): speed of propagation of the pyrolysis zone

\( \rho_i \): density of the \( i^{\text{th}} \) substance

\( f_i \): frequency factor of the \( i^{\text{th}} \) reaction

\( E_i \): activation energy of the \( i^{\text{th}} \) reaction

\( H_i \): heat of reaction of the \( i^{\text{th}} \) reaction

subscripts: \( c \): char, \( g \): volatiles, \( s \): at surface,

\( \infty \): at \( x \to \infty \), \( u \): virgin material

\( T \): temperature

\( V_g \): volatiles velocity

\( c \): specific heat, assumed the same for all species

\( R \): universal gas constant

\( q_s \): heat flux at the interface

\( \lambda \): thermal conductivity

\( x \): measures distance from the interface, as in fig. 2.2
The system of equations 2.1 to 2.6, with the boundary conditions 2.7 to 2.11 was solved by numerical integration [2.3] for the case of a semi-infinite slab of cellulosic material. A theoretical investigation to examine the effects of thermicity in the pyrolysis study has shown that such effect is small. This indicates that the heat source or sink associated with the pyrolysis process of cellulose may be neglected in the energy equation (2.4) without including a significant error in the solution. This assumption led [2.1] to the following closed-form approximate solution:

\[ V_s = \left( \frac{\lambda}{\rho c} \left[ \frac{E}{cRT} \right] + \frac{g}{c} \left\{ E \left( \frac{E}{cRT} \right) - E \left( \frac{E}{cRT} \right) \right\} \right)^{1/2} \]

\[ T_s = T_\infty + \frac{q_s}{\rho c V_s} \quad (2.12) \]

where \( E_1(x) = \int_{-x}^{\infty} \frac{e^{-t}}{t} \, dt, \, x < 0 \) (exponential integral function)

Comparison between the numerical solution and the approximate solution (equation 2.12) for the same set of material properties (Table I) shows good agreement (figures 2.3 and 2.4).

It should be noticed that for a given material this model predicts a unique relationship between the steady speed of propagation of the pyrolysis zone \( V_s \) and the temperature at
the interface ($T_s$). Figure 2.4 shows this relationship for the case of material #10 (Table II).

In a smoldering situation, the steady smoldering will propagate at the speed at which the pyrolysis zone propagates. In order to calculate this steady speed one needs to know the heat flux which is imposed on the pyrolysis zone by some external heat source, or in the case of self-sustained smoldering, by the char-oxygen reaction. The following section is concerned with this part of the analysis, namely the heat generation in the char-oxidation zone.

### 2.1.2 Char-Oxidation Zone

This is the zone where the char produced in the pyrolysis process reacts with the oxygen of the environment and generates heat. The generation of heat per unit area of char is given by:

$$q = r \times Q \text{ [cal/cm}^2 \times \text{sec]}$$  \hspace{1cm} (2.13)

where

$r = \text{reaction rate [gr of char/cm}^2 \times \text{sec]}$

$Q = \text{heat of combustion [cal/gr of char]}$

The reaction rate ($r$) is given by the following expression:

$$r = f_e \cdot p_{O_2}^{1/2} \cdot s \cdot \exp\left(-\frac{E}{RT_s}\right)$$  \hspace{1cm} (2.14)

where:

$f_e$: frequency factor of the char-oxidation reaction

$[\text{gr/cm}^2 \times \text{sec} \times \text{atm}^{1/2}]$
\( P_{O_2} \): oxygen partial pressure at the surface [atm]

\( E_e \): activation energy [cal/mole]

The presence of oxygen at the surface, indicated by \( P_{O_2} \), is depending on two processes; viz., the oxygen consumption in the chemical reaction and the diffusion of oxygen from the environment to the surface. So the reaction rate is also given by the following relation:

\[
    r = \frac{J_{O_2}}{\phi} \tag{2.15}
\]

where:

\( J_{O_2} \): diffusion flux of oxygen [gr/cm\(^2\)s]

\( \phi \): stoichiometric factor [gr of \( O_2 \)/gr of char]; for complete combustion (product \( CO_2 \)) \( \phi = 2.67 \)

The oxygen consumption at the surface creates a gradient of oxygen concentration which induces the diffusion process (Fick's Law). The oxygen will diffuse to the char-oxidation zone, through the inert diluent present in the environment. (\( N_2 \), Ar, or \( He \)) and also through the products of reaction which will be diffusing in the opposite direction. The composition and concentration of the products of reaction is not known. Then as an approximation it is assumed that at least at low reaction rate their effect on the oxygen diffusion process is negligible, so the calculation is performed as if the oxygen were diffusing through the inert diluent
only. It is added to this argument that uncertainties in the kinetics of pyrolysis and char oxidation make refining this aspect of the problem unjustifiable. Then the diffusion equation for the binary mixture of gases is: \[2.4\]

\[
J_{O_2} = -\rho \frac{M_{O_2} M_D}{M^2} D_{12} \nabla X_{O_2} \tag{2.16}
\]

\[M = X_{O_2} M_{O_2} + X_D M_D \]

where:

- \(X_{O_2}\): oxygen mole fraction
- \(X_D\): inert diluent mole fraction
- \(M_{O_2}\): oxygen molecular weight (32)
- \(M_D\): inert diluent molecular weight
- \(\rho\): gas density
- \(D_{12}\): binary diffusion coefficient

The film theory approximation enters at this point to simplify the equation of diffusion. It assumes that the diffusion takes place through a thin boundary layer of gas next to the surface, in which the gradients of concentration and temperature are linear and the properties of the gas inside this layer are equivalent to those evaluated at some average conditions between the surface and some distance bigger than \(\delta\) (where \(\delta = \) thickness of the boundary layer).
Hence:

\[
J_{O_2} = - \bar{\rho} \frac{M_{O_2} M_D}{M^2} D_{12} \frac{X_{O_2, \infty} - X_{O_2,s}}{\delta} \quad (2.18)
\]

where:

- subscript \( \infty \) means far from the surface
- subscript \( s \) means at the surface
- upper bar means average conditions

There is another assumption here, the equation implies that the point of average temperature and average concentration coincide.

Using the perfect gas equation of state \( \bar{\rho} = \bar{M}/RT \) equation 2.18 may be written as:

\[
J_{O_2} = \frac{M_{O_2} M_D}{\bar{M}} \frac{D_{12}}{RT} (P_{O_2} - P_{O_2,s}/\delta) \quad (2.18a)
\]

Then \( P_{O_2,s} \) can be found from equations 2.14, 2.15 and 2.18 giving the following result:

\[
P_{O_2,s} = f(\xi) P_{O_2,\infty} \quad (2.19)
\]

with

\[
f(\xi) = 1 + \frac{\xi^2}{2} - \sqrt{1 + \frac{\xi^2}{2}} - 1 \quad (2.20)
\]
and

\[
\xi = \frac{f_\varepsilon P_{O_2,\infty}^{1/2} \exp\left(-\frac{E_e}{RT_s}\right)}{-\rho \frac{M_2 M_D}{M^2} \frac{X_{O_2}}{\delta \phi} D_{12}}
\]  

(2.21)

Observe that:  \( \xi = \frac{\text{diffusion time}}{\text{chemical time}} \)

the reaction rate can be written now as:

\[
r = f_\varepsilon (f(\xi) \cdot P_{O_2,\infty})^{1/2} \cdot \exp\left(-\frac{E_e}{RT_s}\right)
\]  

(2.22)

Then the heat generated per unit area and per unit time in the char-oxygen reaction (eq. 2.13) is:

\[
q = Q \cdot f_\varepsilon \cdot (f(\xi) \cdot P_{O_2,\infty})^{1/2} \cdot \exp\left(-\frac{E_e}{RT_s}\right)
\]

This heat will be called the heat generated (\(q_g\)), and as it was mentioned before part of it goes to the environment as heat losses (\(q_L\)) and part goes to the pyrolysis zone to sustain the pyrolysis process. The last portion of the heat generated will be called the heat available for pyrolysis (\(q_a\)).

In the previous section it was presented an approximate expression (eq. 2.12) which relates the heat flux imposed at the surface of the pyrolysis zone and the speed at which this zone will propagate through material. This heat imposed (\(q_s\))
will be called heat required for the pyrolysis process.

Notice that \( q_a \) (the heat available) is a function of temperature and \( q_s \) (heat required) is also related to some temperature and speed. The area associated with both heat fluxes is the interface area. Then the matching conditions for the two zones is the temperature at which \( q_a \) and \( q_s \) are the same.

Since \( q_a \) comes from an energy balance which involves losses of heat to the environment:

\[
\text{(total heat available)} = \text{(total heat generated)} - \text{(heat losses)} + \text{(any external source)}
\]

it might occur that \( q_a \) is less than \( q_s \) for all temperatures. In such a situation steady smoldering can not exist, and any initiated smoldering will be a transitory process that eventually will go to extinguishment. In this manner the theoretical model is able to predict conditions at which self-sustained smoldering will not occur. These conditions will be called extinguishment conditions. The boundary between the self-sustained smoldering conditions and the extinguishment conditions will be called extinguishment limit.

It is expected in reality that a flaming limit also exits, however in that aspect the theoretical model is still incomplete and not able to predict any flaming situations.

The experimental part of this thesis examines how the environment properties and composition as well as the physical
properties of the material affect the steady smoldering behavior and the extinguishment limit. To interpret and to compare these results with the theory, several calculations were performed using this theoretical model, applied to the material properties of the experimental samples and the environmental conditions in the experiments. The following section intends to describe the steps of the calculation and presents the predicted behavior of smoldering.

2.2 THEORETICAL CALCULATIONS

2.2.1 Description of the Problem

Figure 2.5 illustrates the situation. A cylindrical sample of cellulosic material is horizontally mounted in the gravity field. The material is smoldering and the smoldering zone moves through the sample at the steady speed V. The characteristics of the environment as well as the characteristics of the material are known. The problem is to calculate the steady speed V and the temperature T_s associated with it. The description of the smoldering element in fig. 2.5 is an approximation of the actual observations, in the sense that the relative dimensions of the char-oxidation zone may vary from one experiment to another.

As suggested in the description of the model, each zone is treated separately to find the heat required (q_s) and the heat available for the pyrolysis process (q_a) as two functions of temperature. Then the matching temperature at the interface is determined graphically at the point where the two functions intersect.
2.2.2 Pyrolysis

It is assumed that the heat flow within the smoldering element is one-dimensional (in the x-direction on fig. 2.5). So the pyrolysis may be considered as moving through a semi-infinite slab of material and the results of section 2.1.1 may be used here.

The numerical integration [2.5] that gives \( q_s \) as function of temperature (fig. 2.3) is available only for one set of material properties (material # 10 - Table II). Then to examine the effect of the properties of the material one finds very helpful the approximate solution given by equation 2.12, which can be written as:

\[
V = \left\{ D f(T_s, \text{kinetics}) \right\}^{1/2}
\]

\[
q_s = (T_s - T_\infty) \rho_\infty V c
\]

where:

\[
D = \frac{\lambda}{\rho_\infty c} \frac{f_e}{\kappa u_\beta}
\]

\( \beta \) is taken as \( 10^{-6} \)

Samples of cotton with different densities are expected to have the same overall kinetics of pyrolysis, so that the function \( f(T_s, \text{kinetics}) \) remains the same for any density. Hence the following relation may be established:

\[
V_i = V_0 \left\{ D_i/D_0 \right\}^{1/2}
\]

(2.24)
\[ q_i = V_i C_i \rho_i (T_S - T_\infty) \]  \hspace{1cm} (2.24)

where \( V_0 \) reference speed at \( T_S \)
subscript 0 corresponds to reference material
subscript \( i \) corresponds to \( i^{th} \) material

Calculations for material # 10 of Table II are available in figures 2.3 and 2.4 (\( D_0 = -722 \text{ cm}^2/\text{sec}^2 \)) and reference [2.1]. With those results and equation 2.24 values of \( q_s \) and \( V \) as functions of temperature were determined for different sets of material properties. These calculations are presented in figures 2.6 and 2.7.

The values of \( q_s \) here obtained must be interpreted as the heat flux that the material requires to decompose at a certain rate and it does not include any heat losses that might occur in the pyrolysis zone. For the complete calculation of smoldering temperature and speed. The heat losses in the pyrolysis zone are neglected, because its average temperature is low and its external area seems small (fig. 2.5). This assumption implies that the pyrolysis process is independent of environment and shape of the sample, and only dependent on its physical properties. This heat losses will increase the required heat for the pyrolysis process and in some situations (small diameter, He-O_2 environments). such effect may be detected. Further discussion of this effect is
presented on the next chapter. Before that more visible effects of environment and diameter shall be considered in the following sections.

2.2.3 Char-Oxidation

According to the analysis of the char-oxidation zone made in section 2.1.2. The environment characteristics and the shape and size of the sample are the necessary information to calculate the generation of heat \( q_g \) as a function of temperature. A little more information and a few assumptions are needed to calculate the heat available \( q_a \) as a function of temperature. For the case considered (fig. 2.5) the calculation procedure was the following.

The parameters \( \bar{M}, D_{12}, \) and \( \delta \) from equation (2.21) have not been determined yet because they depend on the particular situation. In this case they are determined in the following fashion.

Calculation of \( \bar{M} \)

\( \bar{M} \) represents the average value of the mole fraction of the gas within the boundary layer. At \( X \rightarrow \infty \) the value of \( M_\infty \) is known and given by equation 2.16, at the surface \( M_s \) is unknown. In order to get some estimate of \( \bar{M} \) the following assumption is made:

\[
\bar{M} = 0.5 \left( M_\infty + M_D \right)
\]

which implies that at the surface the oxygen concentration is nearly zero and the outcoming gases of the reaction have no effect
The reaction products effect is not included here for the same reasons it was not included in the diffusion equation 2.16.

**Calculation of \( D_{12} \)**

\( D_{12} \) is the binary diffusion coefficient, from references [2.5, 2.7, 2.9, 2.9] for the mixtures of gases used in the experiments.

\[
D_{12} = 5.64 \times 10^{-5} \frac{T^{3/2}}{P} \text{ [cm}^2\text{/sec]}
\]

for \( N_2 - O_2 \)  \hspace{1cm} (2.26a)

\[
D_{12} = 1.95 \times 10^{-4} \frac{T^{3/2}}{P} \text{ [cm}^2\text{/sec]}
\]

for \( He - O_2 \) \hspace{1cm} (2.26b)

\[
D_{12} = 5.358 \times 10^{-5} \frac{T^{3/2}}{P} \text{ [cm}^2\text{/sec]}
\]

for \( Ar - O_2 \) \hspace{1cm} (2.26c)

all evaluated at \( \overline{T} = 1/2(T_\infty + T_s) \)

**Calculation of \( \delta \)**

The boundary layer thickness (\( \delta \)) is determined from heat transfer data under the assumptions of Lewis number equal to unity (\( Le \equiv \alpha/D, \alpha \) thermal diffusivity). Figure 7.10 of reference [2.10] is used to obtain the Nusselt number (\( Nu = d/\delta \)) corresponding to the Grashof number in the boundary layer \( Gr \equiv \rho^2 d^3 \beta_T (T_s - T_\infty)/\mu^2 \).

\( \beta_T \) : coefficient of thermal expansion, for ideal gases \( \beta_T = 1/T \); 
\( \mu \) : viscosity. The following correlations for the data on the reference figure are used to calculate \( Nu \) in the range of interest.
For $5 < Gr < 5000 \ N_u = 1.23(Gr \ Pr)^{0.15}$ \hspace{1cm} (2.27a)

For $0.1 < Gr < 100 \ N_u = 1.1(Gr \ Pr)^{0.15}$ \hspace{1cm} (2.27b)

with $Pr = c_p \mu / \lambda$ Prandtl number assumed equal to $0.7$.

Equation (2.27a) gives $N_u$ for the range of $Gr$ found in $N_2$-$O_2$ and $Ar$-$O_2$ environments, whereas equation (2.27b) gives $N_u$ for the case of $He$-$O_2$ environments.

The properties $\lambda$ and $\mu$ of the gas mixture must be calculated as functions of temperature and composition. In order to perform these calculations empirical and theoretical correlations were employed [2.10, 2.11, 2.12] their description is given in appendix [A2.1].

Once $\bar{M}$, $D_{12}$, and $\delta$ have been determined, the rate of reaction can be calculated (equations 2.20 to 2.22). Equation 2.11 may be rewritten in the following form.

$$\xi = \frac{f_e P_{O_2, \infty}^{1/2} \exp \left(\frac{-E_e}{RT_s}\right)}{A \frac{M_{O_2} M_D}{M_R}^{1/2} \frac{X_{O_2, \infty}}{\delta}}$$ \hspace{1cm} (2.28)

in which $A$ is the corresponding constant in equations 2.26.

The heat generation as function of temperature is given by:

$$q_g = Q f_e \left[f(\xi) P_{O_2, \infty}\right]^{1/2} \exp \left(\frac{-E_e}{RT_s}\right)$$ \hspace{1cm} (2.29)

Figure 2.8 presents calculations of $(q_g)$ as function of temperature for two different situations. Notice that the curves
show three regimes depending on the temperature. At low temperature the chemical time (eq. 2.21) is longer than the diffusion time, so the reaction is limited by the kinetics parameters, and may be approximated to:

\[ r = f_e P_{0, \infty}^{1/2} \exp\left(\frac{-E_e}{TR}\right) \]  

(2.29a)

The two curves in the figure coincide because \( P_{0, \infty} \) is the same for both. At high temperature the chemical time is very short so the process is governed by the diffusion of oxygen to the surface, and the reaction rate can be calculated by:

\[ r = J_{0, \infty} / \phi = A \frac{M_0}{M_R} \frac{M_D}{1} \frac{X_{0, \infty}}{\delta \phi} \]  

(2.29b)

The two curves in the figure are separated in that regime because they correspond to samples of different diameters, and consequently different \( \delta \)'s. The two mentioned regimes meet in the mixed regime in which both processes, diffusion and chemical reactions are equally important, and \( q_g \) is given by equation (2.29).

To finally complete the calculation it is necessary to evaluate the heat available \( (q_a) \). As shown in figure 2.5 part of the generated heat is conducted to the char left and to the pyrolysis zone, and part is transmitted to the environment. Thus,

\[ q_g \pi d^2 = q_a \pi d^2/4 + q_t \pi d^2/4 + q_{\text{losses}} \pi d^2 \]
where: \( \pi d^2 \) is the external area of the char-oxidation zone which is assumed from experimental observations of length \( d \). [Fig. 3.5]; \( q_a' \) is the heat conducted to the char left and will be assumed approximately equal to \( (q_a) \). The area for the char-oxygen reaction and for the heat losses is the same (\( \pi d^2 \)), then,

\[
q_a = 2(q_g - q_{losses}) \tag{2.30}
\]

The heat losses are due to radiation and convection from the char-oxidation zone, they are evaluated as follows:

\[
\text{Radiation } q_r = \varepsilon \sigma T_s^4 \text{ [cal/cm}^2\text{sec]} \tag{2.31}
\]

where

\( \varepsilon \): emissivity of char (taken as 0.9 from [2.1])

\( \sigma \): Steffan-Boltzman constant

\[
\text{Convection } q_a = (T_s - T)h \text{ [cal/cm}^2\text{s]} \tag{2.32}
\]

where \( h \) is the heat transfer coefficient, which is obtained from \( \text{Nu} \equiv \frac{h d}{\lambda} \).

Thus equation 2.30 becomes:

\[
(q_a) = 2(q_g - \varepsilon \sigma T_s^4 - h(T_s - T_\infty)) \tag{2.33}
\]

The calculation of \( (q_a) \) was performed for several environmental conditions and properties to examine their effect.
on the smoldering behavior. Also \((q_a)\) was calculated for samples of
different diameter in air to examine the diameter effect on the final
results. Some of the calculated \((q_a)\) curves are presented in figures
2.8 and 2.9.

2.2.4 Final Calculation

The final calculation consists in plotting together the heat
required \((q_s)\) curve and the heat available \((q_a)\) curve to determine
their intersection. Such a calculation is performed for one
specific case in figure 2.10. The figure suggests the existence
of two solutions because there are two intersection points between
\((q_a)\) and \((q)\). It can be shown that the lower temperature solution
is unstable and the stable solution is at the higher temperature
intersection point. Knowing the temperature at the interface of
the two theoretical zones, Figure 2.4 or equation 2.12 will
give the value of the speed of the smoldering wave for the problem.

It is important to notice, that due to the shape of the \((q_a)\)
curve (as in figures 2.9 and 2.10), when the stable solution
exists it will be in the diffusion limited regime for the heat
generation; and when the \((q_a)\) curve gets smaller (as in figure 2.9)
the two solutions (stable and unstable) will converge to one
point, that represents the extinguishment limit solution which
will be in the mixed regime. This observation predicts that the
steady smoldering behavior will be mostly governed by the
oxygen diffusion process whereas the extinguishment limit will be
more dependent on the chemical process.

Many complete calculations (like figure 2.10) were performed in order to predict the effects of environment and fuel characteristics on smoldering. These results are summarized in the following section.

2.3 THEORETICAL PREDICTIONS

According to the model just described, the smoldering behavior is affected by the material (fuel) physical properties and shape, and by the environmental conditions and composition. Each one of these effects is examined separately.

2.3.1 Effect of the Environment

In order to examine the effect of the environment parameters, fixed diameter and density of the sample are assumed for the computations (those of material # 10). The results are presented graphically in figures 2.11 and 2.12. Figure 2.11 shows the expected dependence of smoldering speed on oxygen mole fraction for the environments examined, and figure 2.12 shows the expected dependence of smoldering speed on oxygen partial pressure for $\text{N}_2-\text{O}_2$ and He-\text{O}_2 environments. Notice that this last dependence is in general small:

$$ v_s \approx (p_{o_2})^{0.38} $$  \hspace{1cm} (2.34)

for both environments.
Figure 2.11 suggests that for oxygen mole fractions between 0.20 and 0.30 the smoldering speeds are about the same for either environment. However the shape of the curves $V_s$ vs $X_{O_2}$ is different for each of them. From the figure the following relations are observed.

$$V_s \approx (X_{O_2})^{0.75}$$  \hspace{1cm} (2.35)

for $he-O_2$

and

$$V_s \approx (X_{O_2})^{1.6}$$  \hspace{1cm} (2.36)

for $N_2-O_2$ and $Ar-O_2$

These dependencies on oxygen mole fractions are stronger than the dependence on oxygen partial pressure, furthermore they are the same kind of dependence that is obtained for the generation of heat in the diffusion limited regime equation 2.29b.

Since

$$\delta \approx 1/p_{O_2}^{0.3} \quad \text{(from eq. 2.27)}$$

Hence

$$J_{O_2} \approx p_{O_2}^{0.3} \quad \text{and} \quad (q_g) \approx p_{O_2}^{0.3}$$

Also

$$J_{O_2} \approx X_{O_2} \sqrt{M} \quad \text{(from equation 2.29b)}$$
which implies an effect due to $\bar{M}$, which is also a function of $X_{O_2}$.

This effect of $\bar{M}$ could explain the shape of the $V_s$ vs $X_{O_2}$ curves. For He-$O_2$ $\bar{M}$ has a wide range of variation and it grows with $X_{O_2}$ which makes the He-$O_2$ dependence of $(V_s)$ on $(X_{O_2})$ the weakest of the three examined. For Ar-$O_2$ $\bar{M}$ does not vary too much but it varies differently than for He-$O_2$, here $\bar{M}$ decreases with $X_{O_2}$ then the curve bends in the opposite direction. For $N_2-O_2$ $\bar{M}$ is almost constant, so the dependence is closer than the others to a straight line.

The effect of the environment in the extinguishment limit is discussed in the next chapter because its calculation involves the use of experimental data to determine the kinetic parameters.

**2.3.2 Effect of the Fuel**

Diameter and density are the fuel characteristics examined. A set of calculations is shown in Figure 2.16 and the obtained results are on Table III. The environment on these computations is assumed fixed (air a 1. atmosphere and $T = 300^\circ$K).

Notice that the solution for each combination of diameter and density (Fig. 2.13) is on the portion of the $(q_a)$ curve, which is at almost constant temperature. Hence the effect of density on temperature for the same diameter (same $q_a$ curve) is expected to be very small, (figure 3.20). However as the density increases
more, material needs to be burned in order to maintain the smoldering propagation, since the temperature does not vary significantly then the speed has to be lower (figure 2.7) figure 3.19.

Decreasing the diameter of the sample, the boundary layer thickness (δ) is also decreased, this will make it easier for oxygen to diffuse to the surface, then the \( q_g \) will be higher and \( q_a \) will also be higher. The intersection point (stable solution) will move to higher heat flux and temperature therefore higher speed (figure 3.19).

In conclusion, the smoldering speed will be increased when the diameter and/or density of the sample is decreased. Temperature is not very sensitive to changes in density but it increases when the diameter is reduced.

The following chapter is concerned with the actual experimental smoldering behavior. The comparison of these predictions with the experimental observations will be then established.
REFERENCES

CHAPTER II


2.4. T.Y. Toong, Combustion Class Notes, MIT, 1975.

2.5. C.A. Garris, Personal communication, May 1976.


CHAPTER III

EXPERIMENTS

Experiments were conducted to determine the effect of environment and fuel characteristics on the smoldering behavior. The purpose of this Chapter is to present and discuss the results of these experiments and their comparison with theoretical predictions. The first sections describe the experimental apparatus and the procedure followed in the tests.

3.1 EXPERIMENTAL APPARATUS

The components of the experimental apparatus are: the pressure vessel, inside which most of the experiments were performed; the voltage recorder, employed to measure temperatures in some of the tests; the radiant heat source and a load cell. The description of each of these elements is presented below.

3.1.1 Pressure Vessel

The main features of the pressure vessel are presented in Figure 3.1. The vessel consists of a steel shell 12.7 mm thick, 91 cm in diameter and 140 cm long, with dome shaped ends and a swinging door. It has two 15-cm diameter windows for visual observation, pipe connections and feedthroughs. A sutorbuilt pump, a Welch pump and a Worthington compressor are available for the purpose of changing the environmental conditions inside the vessel.

The vessel is implemented with a metallic frame with wires to support the samples (Fig. 3.2). This frame has electrical connectors at different locations to install thermocouples and four heating
coils that are used as ignition sources.

3.1.2 Radiant Heat Source

The radiant heat source was designed and built to examine the effect of the heat flux of initiation on the steady smoldering behavior. It consists of three elements (Fig. 3.3): the lamp of 500 watts power (GE # Q500 CL/DC); a cylindrical metallic mirror and a frame to sustain the lamp, the mirror and the sample.

A variac is employed with the radiant heat source to vary its power from 0 to approximately 121% of its nominal power of 500 watts.

3.1.3 Load Cell

This load cell, originally designed in [3.1], consists of a horizontal rectangular frame (6" x 24") placed on a transverse knife edge; and a wheatstone bridge circuit (Fig. 3.4). At one end of the frame is the load platform, where a fine resistance wire (0.001" in-diameter) is strung between the frame and a fixed bar directly below the frame. As the smoldering sample on the load platform decreases its weight, the electrical resistance of the wire also due to the reduction in strain, this change in resistance is monitored by means of the bridge circuit.

Since the material tested has low density (0.06 gr/cm) it was necessary to use large samples and high sensitivity of the load cell in order to measure the mass-loss rate. However the effect of the flow field created around the sample by the combustion process, the bouyancy due to the hot gases inside the material and the electrical noise made unpractical the use of this load cell for this combination
of sensitivity and size of the sample.

3.1.4 Description of the Samples

Cylindrical samples of cotton of different diameters and densities were employed in most of the experiments. Their properties are listed in Tables I and II. In Table II a number is assigned to each combination of diameter and density available, for reference.

In some experiments (initiation) non-cellulosic samples (polyurethane) were tested. Their shape was not cylindrical but more like a vertical wall exposed to the radiant heat of the radiant heat sources. These samples are not listed in Table II.

3.2 EXPERIMENTAL PROCEDURE

The purpose of this section is to describe the principal steps followed in the realization of the experiments. These steps vary a little depending on the objective of the test. For experiments to determine the effect of environmental conditions on smoldering behavior the procedure is the following.

Two or four cylindrical samples of material #10 (in Table II), 60 cm long, are placed the metallic fram as shown by fig. 3.2. Each sample is tied to its corresponding heating coil in the frame to assure contact with it. Fine thermocouples (0.002 in-diameter) pass transverse through the samples, so that the junction is approximately in the center of the sample or in some cases on the surface. The the frame is placed inside the vessel in the position shown by fig. 3.1,
where the samples can be observed from the windows. The vessel's door is closed and sealed with 16 to 24 bolts depending on the conditions of the test. The pumps, the compressor and the pipe connections with bottles of oxygen, Argon, Nitrogen and Helium permit to produce inside the vessel the desired environment characterisitcs of the specific experiment. A small blower inside the vessel provides the mixing of the selected gases.

In these tests the ignition source is the heating coil in contact with the sample. Once the desired environmental conditions have been established and the disturbances due to the blower have disappeared the ignition is induced by turning on the heating coil until smoke or glow appears on the sample – then the initiation source is turned off and the smoldering wave has to support itself. The heat flux of initiation is about 1 cal/cm$^2$ sec in these cases, and the measured features are: the smoldering speed, the temperature history at the point where the thermocouple was installed, the ignition time (time that takes the ignition source to initiate smoldering) and in some experiments the extinguishment time (from ignition to extinguishment).

For the experiments to determine the fuel effect on the smoldering behavior the environment is fixed (ambient air) and the procedure is very much the same except the vessel's door is not sealed but only closed to avoid disturbances in the flow field. The materials used are those in Table II.
For the initiation experiments the ignition source is the radiant heat source and the samples are placed in its frame as in fig. 3.3. In these cases the heat flux of initiation and the ignition time are the measured variables. These experiments are performed in ambient air outside the vessel.

In the first two groups of experiments described (inside the vessel) the experiment is finished when the sample has been completely consumed or transition to flame or extinguishment has occurred.

The measurements and observations are the objectives of the following sections.
3.3 EXPERIMENTAL RESULTS AND DISCUSSION

Depending on the environment and fuel characteristics, three regimes of smoldering were observed. These are steady smoldering, transition to extinction and transition to flame. In this order the steady and non-steady cases are examined.

3.3.1 Description of Smoldering

Figure 3.5 illustrates the main features observed in the steady smoldering combustion of a cylindrical sample of cotton. Most of the samples listed in Table II presented the same qualitative features in their steady smoldering regime. As shown by the figure four regions can be distinguished in the smoldering element, these regions are:

a) The virgin material, ahead of the smoldering front,

b) The pyrolysis zone, which appears as a narrow region of different color (brown), and where the visible gases (smoke) originate from,

c) The char oxidation zone, characterized by its glow due to its high temperature. The gases coming out from this region are invisible. The apparent length of this region is about one diameter of the sample, as it was assumed in fig. 2.5 for the theoretical calculations,

d) The char-left. Material left unburned by the smoldering wave. Its color is generally black and its porosity is very high,
however both color and porosity may vary with the conditions in the test. It retains the cylindrical shape of the virgin material but is hollow. This material left disintegrates very easily, however it does not seem dust to contact, it looks more like a greasy substance that sticks to what touches it. This material left can be reignited.

Fig. 3.6 illustrates other aspects of the smoldering element, namely the boundary layer, the smoke plume and the distribution of temperatures in the element and in the gas phase (smoke) coming out of it. This observation corresponds to the specific case of material #10, (Table II), smoldering in air at atmospheric conditions. The temperature at the center is about 100°C higher than at the surface of the smoldering zone. This implies a higher reaction rate and therefore a higher consumption of material in the center than near the surface of the element, which explains the hollowness of the char-left. Depending on the environment these temperatures are higher or lower. Then the amount of material consumed by the smoldering wave is also different so the porosity and sometimes color of the material left vary.

Fig. 3.7 shows the variation of density across the smoldering element for the same experiment of figure 3.6. The smoldering sample of material #10 (Table II) was extinguished by pressing it between two metallic surfaces. Then it was cut in several small pieces. From the weight of each of those pieces a density was determined for the
region in the original (uncut) sample to which each piece corresponds. The same procedure was followed once more in the same material and conditions. The results of these experiments are represented by the curve in fig. 3.7. This procedure can not be applied for tests in different environments because they must be performed inside the pressure vessel. Measurements of the char-left density after the experiments gave values up to 15% of the density of the virgin material (for low smoldering speed tests) and zero density (for high smoldering speed tests).

Other features of steady smoldering (speed and temperature) were found to change significantly depending on the material (fuel) and environment composition. These effects are discussed in the following sections.

3.3.2 Effect of The Environment on Steady Smoldering

The experimental apparatus described in section 3.1.1 enable us to control the oxygen partial pressure. The oxygen mole fraction and the transport properties of the environment (by changing the inert diluent in the gas mixture). Experiments were conducted on samples of material #10 (Table II) to determine the effect of each of these environment characteristics on the steady smoldering behavior. The results of these experiments were the following.

3.3.2.1 Effect of Oxygen Partial Pressure

Experiments were conducted in environments of $N_2-O_2$ and in He-$O_2$ at constant oxygen mole fraction ($X_{O_2}$) and different partial
pressures ($P_{O_2}$). These results are shown in figures 3.8 (for $N_2-O_2$) and 3.9 (for He-$O_2$). As both figures show, the effect of oxygen partial pressure is very weak, a 100% variation in ($P_{O_2}$) produces only a 20% variation in smoldering speed (within the range of the experiments). Comparison with theoretical calculations (solid lines in the figure) gives good agreement in both environments. The calculations were made for the conditions at which more experimental data was available (Viz. $X_{O_2} = 21\%$ in $N_2-O_2$ and $X_{O_2} = 40\%$ in He-$O_2$). All the results in He-$O_2$ are at high oxygen mole fraction ($X_{O_2} > 30\%$), this is because no steady smoldering was obtained at lower oxygen mole fractions in He-$O_2$ environment.

The temperature at the center of the reaction zone was measured for different oxygen partial pressures in $N_2-O_2$ environments. The results, in fig. 3.10, do not show any appreciable effect of the oxygen partial pressure on the smoldering temperature. Figure 3.11 shows complete temperature profiles across smoldering elements in environments at different oxygen partial pressures. These profiles are very much the same and the small differences could be produced by the error involved in the positioning of the the thermocouple inside the sample. These temperature measurements also agree with theory which predicts very small changes in temperature for small changes in speed (fig. 2.4).

### 3.3.2.2 Effect of Oxygen Mole Fraction

Experiments were conducted to determine the effect of oxygen mole fraction on steady smoldering behavior in Ar-$O_2$, $N_2-O_2$ and He-$O_2$
environments. These results, shown in figures 3.12, 3.13 and 3.14, indicate that the effect is not independent on the inert diluent but in general is stronger than the oxygen partial pressure effect. At low oxygen mole fraction, a 100% change in $X_{O_2}$ produces about 100% change in smoldering speed. At high $X_{O_2}$ the speed becomes less dependent on the oxygen mole fraction and for the case of He-O$_2$ environment the $X_{O_2}$ effect is even weaker than the oxygen partial pressure effect. Comparison with theory indicates good agreement for the low oxygen mole fraction cases, whereas the agreement is not so good at high oxygen mole fractions. This departure of theory and experiments may be due to the assumption made in the calculation to neglect the products of reaction effect (the counter-flow diffusion) and the heat losses in the pyrolysis zone (enthalpy loss in the smoke).

Since changes in smoldering speed due to changes in oxygen mole fraction, are rather large (compared to those due to changes in $P_{O_2}$). A more appreciable change in temperature is also expected according to the theory (fig. 2.4). The variation in maximum temperature due to variations in oxygen mole fraction is shown by figure 3.15 for the case of N$_2$-O$_2$ environments at 15-5 cm Hg of oxygen partial pressure. The effect is also clear in Fig. 3.16 which shows the temperature profiles across the smoldering zone for different oxygen mole fractions. The temperature at each point is higher oxygen mole fraction. Notice that there is some symmetry in these profiles (about the maximum temp-
erature, but in the lower temperature regions), the same symmetry is observed in figure 3.11. Is this observation which leads to the assumption (in the calculations) that the heat conducted to the char left and the heat conducted into the pyrolysis zone are about the same (equation 2.30).

3.3.2.3 Effect of the Inert Diluent

In order to examine the effect of the inert diluent (which changes the transport properties of the environment) the results obtained in Ar-O<sub>2</sub>, N<sub>2</sub>-O<sub>2</sub> and He-O<sub>2</sub> environments are compared.

Figure 3.9 (speed vs P<sub>O<sub>2</sub></sub> for He-O<sub>2</sub>) includes some experiments made in N<sub>2</sub>-O<sub>2</sub> environments at conditions similar to those of experiments in He-O<sub>2</sub>. The figure indicates that the smoldering speed is a little faster in Helium than in Nitrogen. It also shows that the dependence of smoldering speed on oxygen partial pressure is the same for both environments. Notice that contrary to the N<sub>2</sub>-O<sub>2</sub> data in figure 3.8, the He-O<sub>2</sub> results occupy a very narrow region in its figure (3.9). This is because steady smoldering in He-O<sub>2</sub> is not as dependent on the environmental conditions as it is in N<sub>2</sub>-O<sub>2</sub> environments. This observation becomes more clear in figure 3.12 where the comparison is established for the dependence of smoldering speed on oxygen mole fractions. This figure shows large variations in speed in environments of Ar-O<sub>2</sub> and in N<sub>2</sub>-O<sub>2</sub> while for He-O<sub>2</sub> the speed is high but less variable with oxygen mole fraction. A possible explanation for this behavior was suggested in section 2.3.1 of the previous chapter.
where the effect of $\bar{M}$ (molecular weight of the environment) on this oxygen diffusion process was examined. However, effect does not fully describe the observed behavior. In the actual case (experiments) the counterflow diffusion flux of products of reaction (neglected in the calculations) is present, and for the case of Helium must be larger than in Ar–O$_2$ or N$_2$–O$_2$ because helium mixtures have higher diffusion coefficients, and that might be the reason why the departure of experiment from the expected behavior is larger in He–O$_2$ than in Ar–O$_2$ or N$_2$–O$_2$ environments.

The effect of the inert diluent on the temperature of the smoldering zone is shown in figure 2.4 in which the theoretical and experimental temperature-speed relationship are compared. As the figure indicates there is no appreciable effect on this relation due to the inert diluent. The experiments seem to agree relatively well with the theory regardless of the environment in the experiment. This observation indicates that helium does not affect the pyrolysis process of cellulose.

Other aspects of smoldering change because of the inert diluent. The visibility on a He–O$_2$ experiment is very poor compared to a similar experiment in N$_2$–O$_2$. This means the sample can be observed more clearly in a N$_2$–O$_2$ test than in a comparable He–O$_2$ test.

The higher viscosity of He–O$_2$ mixtures compared to N$_2$–O$_2$ ones makes the smoke plume remain laminar for a longer distance and the boundary layer to be much thicker (three times thicker) in He–O$_2$ than in N$_2$–O$_2$ environments (for N$_2$–O$_2$ $\delta \approx 0.2$ cm and the plume becomes
turbulent a few diameters above the sample).

Other environmental parameters, such as relative humidity (RH) and ambient temperature ($T_\infty$), which are not controllable, also have their effect on the smoldering behavior. This effect is too small to be measured (same order as experimental uncertainty) but its presence is evident from the comparison of the scatter in the data points obtained in different environments. For Ar-$O_2$ and He-$O_2$ environments, which come from bottles (RH≈0), the scatter is not as big as in $N_2-O_2$ tests, in which air from ambient (RH≠0) is always present. This scatter is particularly high in experiments made in plain air ($X_{O_2} = 0.21$, Fig. 3.8). This scatter which is not present in the Ar-$O_2$ and He-$O_2$ results may be due to the effect of relative humidity in the environment of the test.

A very important effect of the inert diluent is found in the non-steady smoldering regimes. However in order to maintain the continuity of the presentation for steady smoldering, its discussion is completed with the effect of fuel characteristics in the next section. The non-steady cases will follow in a later section.

3.3.3 Effect of Fuel Characteristics

Experiments were conducted using cylindrical samples of cotton, with different densities and diameters, in air at atmospheric conditions (inside the pressure vessel). The results of these experiments are tabulated on Table II.

Comparison between these experimental results and the theoretical
calculations (approximate solutions, equation 2.12) listed in table III is given in figures 3.18, 3.19, 3.20. Figure 3.18 examines the correlation proposed in reference [3.2] which states that

\[ V_s \approx (1/\rho d) \]

As illustrated by the figure the experimental results have the tendency to follow this relationship, however there is some scatter in the data points. The theory predicts that the relation is not a unique one, and it depends weakly on the density of the material. This comparison may explain some of the scatter observed in the data points, for which the effects of ambient temperature and relative humidity must be also responsible.

In Figure 3.19 the effects of density and diameter are separated. Comparison between experiments and the theoretical approximate solutions gives a qualitatively good agreement, in the sense that theory describes the observed dependences of speed on density and diameter, even though the observed smoldering speed is faster than the calculated one (≈1.4 to 1.7 times faster). The comparisons hold between the approximate solution and the exact solution, where this available (material #10, figures 3.8, 3.9, 3.12, 2.13, 3.14 and the calculation itself, figure 2.10 and figure 2.11) indicates that the exact solution would be also a little faster than the approximate one. So that the exact theoretical description of the "\( \rho \)" and "\( d \)" effects might be closer to the experimental observations.
Figure 3.20 shows the temperature dependence on density and diameter, and its comparison with theoretical predictions. The agreement between the two is not very good, and this may be due to the uncertainty in the value of the interface temperature (the calculated $T_s$). The surface temperature for some of the tests have been included in the figure (dashed symbols) to show the wide range of temperatures present in each actual smoldering element (more than 100°C in the worse case).

A number of experiments were conducted in order to determine the combined effects of density, diameter and partial pressure of oxygen (in air) on the steady smoldering speed. These results are presented in figure 3.21. They show that the effect of oxygen partial pressure is small, and about the same regardless of density, diameter, oxygen mole fraction and inert diluent (from figures 3.8 and 3.9).

To conclude, from all the comparisons made (up to here) between theoretical calculations and experimental observations, the model has been able to describe the main features of steady smoldering for the cellulose samples employed in the tests. However not in every case the predicted steady behavior was observed. Non-steady smoldering was observed for certain combinations of environmental conditions and fuel characteristics. The discussion of this observation is presented in the following section.

3.3.4 Non-Steady Smoldering Regimes

The non-steady smoldering regimes are transient to extinction.
and transition to flaming. Both transient behaviors depend on the environmental conditions and composition, density and diameter of the sample and the heating flux of initiation.

Experiments were conducted using samples of material #10 (Table II) to determine, in an environmental condition ($P_{O_2}-X_{O_2}$) map, the regions in which the steady and non-steady smoldering regimes occur. The procedure followed in these experiments is the same as described in section 3.2 to determine the effect of the environment. These tests were performed in $N_2-O_2$ and He-$O_2$ environments, their results are presented in figures 3.22 (with other results reported in literature [3.2]) and 3.23 and also discussed in the following sections. Notice that the heat flux of initiation in all these tests is constant (section 3.2), the effect of this parameter is examined and discussed in a later section.

3.3.4.1 Transition to Extinguishment

Extinguishment was more extensively examined than flaming, mainly because it does not destroy the experimental apparatus (as flaming does), and comparison between theory and experiments can be established.

According to the experimental observation (figures 3.22 and 3.23) there is a limit between the steady smoldering and the transition to extinguishment conditions. The location of this limit and the transient behavior of the smoldering wave is very different in $N_2-O_2$ and He-$O_2$ environments.
In \( \text{N}_2-\text{O}_2 \) environments (figure 3.22), extinguishment occurs at oxygen concentrations lower than in ambient air. The initial speed of the smoldering wave is about 0.7 cm/min, and after some time (up to 25 minutes) the smoldering propagation stops and no smoke is originated in the sample. This initial speed decreases slowly at first and faster in the last minutes. The material left is not as black as in the steady case and has some spots retaining the original color, which is the material that was not pyrolyzed by the smoldering wave.

In \( \text{He-} \text{O}_2 \) environments (figure 3.23), extinguishment occurs at oxygen concentrations higher than in ambient air. The initial smoldering speed is much faster than in \( \text{N}_2-\text{O}_2 \) (2 cm/min in \( \text{He-} \text{O}_2 \)) and the time between initiation and extinguishment is very short (no longer than 5 minutes). The char-left is reduced to a minimum, which is only present near the point where extinguishment occurs (in the sample).

According to theory, transition to extinguishment occurs when the heat generation \( (q_g) \) in the char-oxidation zone is not enough to provide the heat required for the pyrolysis process and overcome the heat losses. Theoretically the extinguishment limit is at the conditions for which the heat available \( (q_a) \) and the heat required \( (q_s) \) curves intersect at only one point, which happens to lie in the kinetic limited regime for the heat generation, hence it is highly dependent on the kinetic parameters of the char-
oxidation reaction. Even though the model does not describe any transient behavior, it does predict the conditions at which steady smoldering can not be self-sustained. Therefore one should be able to determine the kinetic parameter of the char-oxidation reaction from the knowledge of the experimental extinguishment limit. The frequency factor of this reaction \( f_e \) was determined in [3.2] from the knowledge of the extinguishment limit in \( \text{N}_2-\text{O}_2 \) environments (figure 3.22) by trial calculations of the theoretical limit (using the approximate solution for the pyrolysis process). The result and other recommended parameters are listed in Table I.

With the kinetic parameters in Table I the calculation was performed to predict the extinguishment limit in \( \text{He}-\text{O}_2 \) environments. Comparison between this calculation and experiments gives an important difference (figure 3.23). Then a new calculation was performed with a different frequency factor \( f_e = 10^7 \) instead of \( f_e = 10^5 \) of [3.2]. The new curve (in the same figure 3.23) is in a relatively better agreement with experiments. This is not good either because the experimental limit shows less sensitivity to oxygen partial pressure than the calculated limit. In order to find a possible explanation for this result some of the theoretical assumptions must be revised.

As discussed for the steady state case the heat losses in the pyrolysis zone, and neglected in the calculation, may become very important when helium is the inert diluent. The heat
capacity, viscosity, thermal conductivity and diffusion coefficient are larger in He-O\textsubscript{2} mixtures than in Ar-O\textsubscript{2} and N\textsubscript{2}-O\textsubscript{2}. The calculated convective losses are always higher in He-O\textsubscript{2} than in N\textsubscript{2}-O\textsubscript{2} and Ar-O\textsubscript{2} but the most important effect is probably due to the diffusion coefficient and the Grashoff number. The boundary layer observed (in the smoke) and calculated is about three times larger in He-O\textsubscript{2} than in N\textsubscript{2}-O\textsubscript{2} and Ar-O\textsubscript{2}, also the diffusion coefficient in Helium mixtures is larger than in N\textsubscript{2} or Ar mixtures. This indicates that in He-O\textsubscript{2} environments the volatile products in the pyrolysis process will diffuse faster to the environment carrying enthalpy with them, representing an important heat loss that has not been taken into account for the calculation. If these losses were included in the analysis they would increase the heat requirement for the pyrolysis and the steady smoldering calculation would only be slightly modified (figure 3.24a, b), because the intersection point will be at almost the same temperature as it is with the no-losses heat required curve. As illustrated by figure 3.24b, the modification will be larger at higher temperature solutions. The modified smoldering speed would be slower than with no-losses which suggests that the agreement between theory and experiments for the steady state regime at high smoldering speeds might be better if these losses were considered. These heat losses in the pyrolysis zone will also determine a different extinguishment limit.
As shown in figure 3.24c, the no-losses analysis predicts steady smoldering when the actual case might be extinguishment.

The estimated heat loss necessary to bring the calculated extinguishment limit for He-0\textsubscript{2} to the experimental limit position is about 1.2 cal/cm\textsuperscript{2}sec which is the same value obtained for the heat loss in the char-oxidation zone at the same environmental conditions without taking into account the enthalpy associated with the products of reaction. The observed amounts of smoke in He-0\textsubscript{2} test suggested that this amount of heat loss can be easily attained in the pyrolysis zone.

The effect of helium, which increases the heat losses, has been observed before in [3.3]. The ignition time for a given material with a given heat flux imposed, is longer when the atmosphere is He-0\textsubscript{2} than when it is N\textsubscript{2}-0\textsubscript{2}. The same effect is suggested by the fact that extinguishment occurs faster (5 min.) in He-0\textsubscript{2} than in N\textsubscript{2}-0\textsubscript{2} (25 min) even though the heat flux of initiation was the same in both experiments.

Extinguishment is also observed for samples of low density and small diameter at conditions at which theory predicts steady smoldering. The initial speed in these cases is as fast (as in He-0\textsubscript{2}) as it corresponds to small diameter samples and the transition to extinguishment is abrupt. As it the He-0\textsubscript{2} case the small diameter increases the heat losses to a point in which steady smoldering can not longer propagate.
Notice that in determining the frequency factor for the char-oxidation reaction the approximate solution was employed. Comparison between the approximate and exact solution figure 2.3 indicates that the actual frequency factor must be a little higher than recommended. According to calculations the steady smoldering speed is not sensitive to the frequency factor and calculations for different values of $f_e$ give the same result (figure 3.24d).

3.3.4.2 Transition to Flaming

Transition to flaming was allowed only a few times due to the considerable damage that it does to the equipment. Figures 3.22 and 3.23 indicate the conditions at which this transient behavior was found. Figure 3.22 (in $N_2-O_2$) suggests the existence of a flaming limit and comparison of this limit with the results in Figure 3.23 (for $He-O_2$) shows the effect of helium on that limit.

Contrary to the behavior in the transition to extinguishment, near flaming conditions, smoldering behaves very much the same in either of the three environments tested, presenting the following features: the smoldering temperature and speed are relatively high ($\approx 1100^\circ K$ and 3 to 4 cm/min); the char-oxidation zone is very bright and little flashing spots (fig. 3.5) are coming out of it regularly; there is no char-left, all the material is consumed (Fig. 3.17)
by the smoldering wave. These conditions will be called near-flaming conditions and their characteristics are also present in the transient behavior before flaming occurs.

The transition to flame can be observed in conditions at the flaming limit shown in figure 3.22. For conditions beyond (to higher oxygen concentrations) this limit the transition to flame occurs very fast and smoldering has a very short duration (a few seconds). This transition is violent, the flame spreads all over the sample which then is burned all at once, while the flame continues propagating to the rest of the samples and the electrical connections inside the pressure vessel.

The transition to flame can be also obtained from near-flaming conditions by blowing on the sample. This flame is less violent and caused relatively less damage when it occurred in the He-O₂ environments.

Transition to flame seems to occur when the smoldering speed and temperature are within certain values (3.5 to 4 cm/min and \( \approx 1100^\circ K \)). Since the oxygen mole fraction and partial pressure at which these speed and temperatures are attained for the smoldering wave, depend upon the inert diluent in the environment, according to the steady state results, helium will shift the flaming limit to higher oxygen concentrations. The results in figure 3.23 agree with this conclusion, more oxygen is needed to get spontaneous flaming in He-O₂ than in N₂-O₂ or Ar-O₂ environments.
In these tests as in the extinguishment ones, the heat flux of initiation is always the same. When this heat is decreased smoldering may not even occur, when the heat is increased flaming may be obtained (both situations within the steady smoldering regime). This indicates an effect on the non-steady smoldering behavior due to the heat of initiation. This effect and other observations regarding the initiation of smoldering are discussed in the following section.

3.3.4.3 Smoldering Initiation Experiments

Since the ignition source employed in most experiments is a heating coil, for which the heat flux can only be estimated, and which is also a piece of equipment with limited life and had to be replaced several times. It was necessary to determine the possible effect of the heat flux of initiation on steady smoldering. In order to do this a radiant heat source was designed and built (described in Section 3.1.2, figure 3.3). Experiments were conducted on samples of material # 10 (Table II) in air at atmospheric conditions, outside the pressure vessel. The variac permitted to obtain relatively wide variations in heating flux of initiation (from the lamp or from the heating coil).

Four experiments were made (the same day, to avoid other effects), in three of them the ignition source was the lamp and in the other the ignition source was the heating coil as it is normally used for other experiments. So, four different heating
rates of initiation were tested. The external heat flux is
turned off when smoke results from the sample. The result of these
experiments shown in figure 3.25 indicates that at least small
variations of the heating rate of initiation have no effect on the
steady smoldering behavior.

A second group of experiments was conducted in material # 10
and other cellulosic and non-cellulosic materials to explore
their capability of smoldering and the effect of the initiation
heat flux on the different smoldering regimes. The sample did
not have any specific shape, (they were placed in the radiant
heat source at certain distance from the lamp (ignition source)).
The lamp is used to ignite the material and is turned off once
ignition (appearance of smoke, glow or flame) has occurred. The
heating rate of initiation is estimated from the knowledge of the
power of the lamp (500 watts) and the distance between the sample
and the filament of the lamp. Thus the heat flux at 1" from the
filament is about 1.4 cal/cm²·sec for 100% power of the lamp. The
results of these experiments are presented in Table IV. They also
show that flame can be obtained for sufficiently high heat fluxes
of initiation in steady smoldering regimes.

A third group of experiments was conducted in material # 10
(Table II) and samples of polyurethane to determine the effect of
heat initiation in the ignition time and to compare the behaviors of
the two materials. The procedure for this experiment is the same as
for the second group, but in this case the time from turning on the lamp to the appearance of any sign of ignition was measured. The results of this group of experiments are presented in Figure 3.26. They show that polyurethane needs a very low initiation heat to be ignited, however no steady smoldering was observed in this material. It stops burning when the heat source is suppressed, and a low external heat flux can help it to sustain the combustion process.

It should be mentioned that when using the radiant heat source it was necessary to impregnate the surface of the sample with char in order to increase its absorptivity to the radiant heat, otherwise very poor.

3.3.4.4 Induced Transition

A number of experiments were conducted to explore the possibility of inducing transition from one smoldering regime to another by alternating the ambient conditions at which the original smoldering regime was obtained. Particular interest was given to the induced transition from steady smoldering to extinguishment.

Transition from steady smoldering to flaming was obtained: (1) by blowing on a sample which is smoldering at near flaming conditions; (2) when the heating rate of initiation is high, even though the environmental conditions correspond to the steady smoldering regime.
Transition from flaming to steady smoldering was obtained:
(1) by blowing on the sample in which flaming was induced by a high heating rate of initiation (i.e. a match) and the conditions are that of steady smoldering regime (in flaming conditions this does not occur); (2) by reducing the total pressure inside the pressure vessel, observed in some cases in which accidental transition to flaming had occurred. Unfortunately in these cases the necessity of stopping the flame as quickly as possible did not permit any measurements to be taken.

Transition from steady smoldering to extinction was obtained: (1) by reducing the total pressure inside the pressure vessel (with vacuum pump) until the environmental conditions were in the extinction regime region of the environmental conditions map of figure 3.22 (for N$_2$–O$_2$ environments). This process could be represented by a vertical line (constant X$_{O_2}$) in the mentioned map; (2) by reducing the oxygen mole fraction, injecting gas (pressurization) it was observed that the inert gas selected has an effect on the oxygen mole fraction required to obtain the extinguishment. The description and results of these pressurization experiments are the following:

Smoldering is initiated and reaches steady state at some selected initial environmental conditions in N$_2$–O$_2$ environments. As shown in figure 3.27 (by i.c.), then the inert gas is injected inside the vessel in steps (represented in figure as segments)
until extinguishment conditions are found (represented by (□)). The oxygen concentration required to obtain extinguishment is higher when the injected gas is He than when it is N₂, also, the time required for the injection and the transient time are much shorter with He than with N₂.

The effect of helium increases as its relative amount in the mixture increases. In oxygen enriched atmosphere the transition from steady smoldering to extinguishment is much closer to the original conditions, (i.e.) figure 3.27, when He is injected than when N₂ is injected. So less amounts of gas are needed when He is employed as inert diluent injected.

This effect of helium, which is probably due to the higher transport properties it induces in the gases mixture of the environment, might have an important application in extinguishing smoldering fires. Such applications are examined in the following chapter.
CHAPTER III

REFERENCES


CHAPTER IV

EXTINGUISHMENT OF SMOLDERING FIRES

Experimental observations described in sections 3.3.4.1 and 3.3.4.5, suggest the pressurization with a non-reacting gas as a method to extinguish smoldering fires in enclosures. The purpose of this chapter is to examine: the feasibility of this method, its comparison with existing extinguishment methods, and its effect on other types of fires (i.e. flaming or explosion).

4.1 Method of Extinguishment

The application of the proposed method, as well as the application of any other method, always depends on the specific situation in which it is going to be employed. Therefore a general description of the mechanism and its requirements is found more useful than a detailed calculation for one specific case. However, in order to illustrate the applicability of the method a particular example is discussed in Appendix A-4-1. The description of some possible procedures are the following:

4.1.1 Pressurization With Non-Reacting Gas

The purpose of pressurizing with a non-reacting gas is to make the environment of the fire incapable of sustaining smoldering combustion (extinguishment regime environment). According to experiments the same result would be obtained by depressurization of the environment (section 3.3.4.5). However depressurization requires
pecial equipment (vacuum pumps) and special construction of the enclosure (it must be able to support and sustain vacuum), and most important of all depressurization is not a desirable procedure when the habitability of the environment is required. On the other hand pressurization with the gases studied (\( \text{N}_2 \) and \( \text{He} \)) requires only the storage (bottles) or suitable supply of the non-reacting gas from other sources, and being non-toxic, it allows the presence of personnel in the enclosure during and after the time of extinguishment, which is the most important advantage of this method over others studied.

Experiments have shown (Fig. 3.27) that the amount of non-reacting gas necessary to obtain extinguishment by pressurization depends on the initial conditions of the environment and the gas which is injected. Comparison between results obtained with Helium and Nitrogen indicates that helium is more effective than nitrogen, in the sense that: less amount of gas is needed, the transition to extinguishment is faster and less time is also required for the injection of helium due to its properties.

The pressurization with inert gas does not imply high increments in pressure, according to experiments (section 3.3.4.4) and calculations (appendix A.4.1), for a situation in which the initial environment in air at 1 atm the final pressurized atmosphere will probably be of 1.3 to 1.4 atm in pressure, which does not require very special construction of the enclosure. This makes the method applicable to enclosures like: ships, aircrafts, submarines, space vehicles, storage rooms, and
computer facilities, in which also the habitability of the environment is important.

Some of the advantages of pressurizing with non-reacting gas have been already mentioned, others would be the fact that it does not affect equipment or stored material, there are no cleaning problems and best of all it is expected to be fast and effective in extinguishing the smoldering fires. The method does not seem to have critical disadvantages, however it might become impractical and very expensive when the size of the fire is much smaller than the size of the enclosure, or when the fire is not inside any enclosure as it is in the case for a dwelling smoldering fire. In these cases it might be possible to create the appropriate extinguishant environment around the fire by spraying the inert gas on it as it is done with a conventional CO₂ manual extinguisher. This possibility is discussed in the following section.

4.1.2 Extinguisher With Inert Gas

Experiments were conducted to compare the effectiveness of helium extinguishers with CO₂ extinguishers and water. Three similar piles of cotton of about 12 cms diameter were placed on a frame (fig. 4.1) at ambient conditions. Each of them was heated with a match in different locations until most of the external part of the material was smoldering. Then in one of them a jet of helium was applied for a few sections, extinguishment occurred and no reignition was observed. In the second sample the manual CO₂ extinguisher was
applied, the white material covered all the surface of the sample but after a few minutes of its application it disappeared and smoke was observed again and the sample returned to its original smoldering process. The third sample was treated with water. The smoldering combustion stopped immediately and no reignition was possible in the wet material.

According to these results the inert gas (helium) extinguisher is effective in extinguishing small smoldering fires, in which the CO$_2$ extinguisher might fail. As is was mentioned in the first chapter, other fire extinguishant procedures have been uneffective in smoldering fires [1.3, 1.4]. The comparison of the proposed method with these other methods is established in the following section.

4.2 Comparison of Inert Gas with Other Extinguishant Procedures

As mentioned before the possible advantages or disadvantages of one method over the others is a function of the specific problem, but in general the practical qualities and limitations of the examined extinguishment procedures are the following:

A) Water: is the most effective extinguishment media for smoldering fires, however it is not desirable when it may occasion damage to equipment or stored materials. It must not be used when electrical active systems are involved in the fire.

B) Foams: are good in flaming fires and sometimes in deep
seated smoldering fires \[4.1\]. They have the same limitations that water has because they contain water in their composition. They also require evacuation of personnel from the room of the fire because they are irritating to the skin. They also need very heavy equipments, trucks, compressors, foam flooding systems.

C) \( \text{CO}_2 \): is also good in flaming fires but very ineffective in smoldering fires \[4.2\]. It is also toxic so it requires evacuation of personnel.

D) Dry Chemicals: salt dusts which are good in flaming fires, but can not diffuse to deep seated smoldering fires. It is difficult to clean equipment after they have been applied. They are not recommended for fires in which electronic equipment is present \[4.3\]. They require special systems which make them expensive.

E) Halon 1301 (\( \text{CF}_3\text{Br} \) (monobromothrifluoromethane): it is effective most of the time. How it actually works is not known yet \[4.4\]. It requires to be in a concentration of 5% which is lower than the required concentration for helium (\( \approx 19\% \)) in the experiments. But it seems to be slower and more expensive than helium. It also may become unstable and toxic \[4.5\].
According to these descriptions, when water can not be applied, the real competitor of helium seems to be Halon 1301. However a rough comparison in costs [4.6] of the two gases with respect to the volume of room to which they would be able to provide extinguishment conditions, indicates that halon 1301 is four times more expensive then helium. Also due to its properties and composition halon 1301 requires a heavier and more sophisticated storage and distribution system than helium, which furthermore is expected to diffuse faster then the heavy molecules of halon 1301 (according to experiments and reported data on Halon 1301 [4.7]). This however must be regarded as an approximate comparison, according to [4.8] a full size test with helium (not possible with the available equipment) is necessary to prove its effectiveness and presumed advantages over Halon 1301.

The results obtained with helium correspond to cellulosic materials, which are a good part of the combustable materials present in fires type A (common combustibles: wood, paper, cloth, etc) and in fires type C (electrically initiated fires). However in many of the situations mentioned (enclosures) there are other materials present (wook, polyurethane, upholstered funiture, etc) which also smolder, and in which the smoldering mechanism might not be the same as studied here. Then further work is necessary before the extinguishment method proposed is actually applied in a real fire.
4.3 Effect of Non-Reacting Gases on Flaming and Explosions

4.3.1 Flaming Fires

Inert gases have been used before to extinguish flaming fires in ships [4.9] and the method has been found effective and non-damaging to equipment. Investigations have been conducted [4.10 and 4.11] to study the pressurization with N\textsubscript{2} as an extinguishment procedure for fires classes A and B (liquid fuels). The results of those investigations indicate that the method is also fast and effective in flaming fires.

According to experiments described in section 3.3.4.2 helium-oxygen atmospheres are effective in reducing the possibility of transition to flaming.

4.3.2 Explosions

The NFPA [4.12] recommends the use of inert gases to reduce the hazards of explosions in tanks where combustible gases or liquids have been stored. The inert gas reduces the possibility of explosions.
CHAPTER IV
REFERENCES

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Section 17, Chapter I.

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Extinguishers, pp. 408-5, 408-10.

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Chapter V, pp. 4-5.


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personal communication with "Fire Equipemnt, Inc." Medfore,
Mass., (June 1976). Information on Helium prices obtained
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4.7 DuPont Halon 1301 Fire Extinguishant Publication #B-29C
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Section 18, Chapter I, pp. 18-2, 18-13.

4.9 Fire Extinguishing with Inert Gas" The British Motor Ship,
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TABLE I

Values of Material Properties Employed in the Calculations

For the Pyrolysis Study:

(c) Cellulose specific heat = 0.20 cal/°K gr

(λ) Cellulose thermal conductivity = 1.2 \times 10^{-4} \text{ cal/cm}°\text{K sec}

(T_∞) Ambient temperature = 300°K

For the Char-Oxidation Study:

(E_e) Activation Energy = 44 K cal/mole

(f_e) Frequency factor = 10^8 \text{ gr of char/cm}^2 \text{ sec atm}^{1/2}.

(Q) Heat of combustion of char = 7700 cal/gr of char

(Ø) Stoichiometric ratio = 2.67 gr of O_2/gr of char

(ε) Char emissivity = 0.9
TABLE II

List of cotton samples employed in the experiments, and their smoldering steady speed and temperature in ambient air, (all cylindrical samples) with properties in Table I.

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<th>mat. #</th>
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<th>d cms</th>
<th>$V_s$ cm/min</th>
<th>T°K</th>
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<td>0.537</td>
<td>0.41</td>
<td>0.51</td>
<td>985</td>
</tr>
<tr>
<td>19</td>
<td>0.514</td>
<td>0.595</td>
<td>0.395</td>
<td>-</td>
</tr>
<tr>
<td>20</td>
<td>0.06</td>
<td>3.5</td>
<td>0.68</td>
<td></td>
</tr>
</tbody>
</table>
TABLE III

Results of the theoretical calculations for cylindrical samples of Cotton smoldering in air at ambient conditions (using the approximate solution).

<table>
<thead>
<tr>
<th>d(cm)</th>
<th>[gr/cm³]</th>
<th>T_s °K</th>
<th>c_s [cal/cm²s]</th>
<th>s [cm/min]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.14</td>
<td>0.06</td>
<td>1176</td>
<td>0.525</td>
<td>3.</td>
</tr>
<tr>
<td>0.14</td>
<td>0.3</td>
<td>1149</td>
<td>0.97</td>
<td>1.142</td>
</tr>
<tr>
<td>0.14</td>
<td>0.5</td>
<td>1140</td>
<td>1.15</td>
<td>0.821</td>
</tr>
<tr>
<td>0.22</td>
<td>0.06</td>
<td>1117</td>
<td>0.355</td>
<td>2.17</td>
</tr>
<tr>
<td>0.22</td>
<td>0.3</td>
<td>1099</td>
<td>0.66</td>
<td>0.826</td>
</tr>
<tr>
<td>0.22</td>
<td>0.5</td>
<td>1090</td>
<td>0.77</td>
<td>0.585</td>
</tr>
<tr>
<td>0.38</td>
<td>0.06</td>
<td>1052</td>
<td>0.205</td>
<td>1.36</td>
</tr>
<tr>
<td>0.38</td>
<td>0.3</td>
<td>1037</td>
<td>0.4</td>
<td>0.542</td>
</tr>
<tr>
<td>0.38</td>
<td>0.5</td>
<td>1031</td>
<td>0.475</td>
<td>0.39</td>
</tr>
<tr>
<td>0.54</td>
<td>0.5</td>
<td>990</td>
<td>0.33</td>
<td>0.287</td>
</tr>
<tr>
<td>0.54</td>
<td>0.3</td>
<td>998</td>
<td>0.275</td>
<td>0.394</td>
</tr>
<tr>
<td>0.54</td>
<td>0.06</td>
<td>1009</td>
<td>0.14</td>
<td>0.9872</td>
</tr>
</tbody>
</table>
TABLE IV

Results of the initiation experiments on different materials in air at ambient conditions. The heat flux of initiation is provided by the radiant heat source, and is imposed on the sample surface until smoke or glow or flame is observed then is turned off. The heat flux is varied by varying the power of the lamp with the variac.

<table>
<thead>
<tr>
<th>Material</th>
<th>Heat Flux Cal/cm$^2$</th>
<th>Observation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cotton (roving)</td>
<td>from 0.75 to 1.25</td>
<td>steady smoldering</td>
</tr>
<tr>
<td></td>
<td>1.7</td>
<td>flame</td>
</tr>
<tr>
<td>Ash free analytical</td>
<td>0.94</td>
<td>only smoke</td>
</tr>
<tr>
<td>filter pulp</td>
<td>1.1</td>
<td>transition to extinction</td>
</tr>
<tr>
<td></td>
<td>1.4</td>
<td>flame</td>
</tr>
<tr>
<td>Solka flock</td>
<td>0.68</td>
<td>transition to extinction</td>
</tr>
<tr>
<td></td>
<td>0.9</td>
<td>self-sustained smoldering</td>
</tr>
<tr>
<td>Avicel PH-101</td>
<td>1.7</td>
<td>nothing happens</td>
</tr>
<tr>
<td>Princeton</td>
<td>0.68</td>
<td>only smoke</td>
</tr>
<tr>
<td></td>
<td>from 1.1 to 1.4</td>
<td>self-sustained smoldering</td>
</tr>
<tr>
<td></td>
<td>1.7</td>
<td>flame</td>
</tr>
</tbody>
</table>
APPENDIX A2.1
ENVIRONMENT PROPERTIES

The following theoretical and empirical correlations were employed to calculate the viscosity (\(\mu\)) and thermal conductivity (\(\lambda\)) of the gas mixture in the environment as a function of temperature and composition.

A) Properties for pure gases.

**Viscosity**: (from ref. [2.10])

for He: \(\mu = 3.7 \times 10^{-4} + (T - 773) \times 3 \times 10^{-7} \text{ [gr/cms]}\)

for O\(_2\): \(\mu = 3.5 \times 10^{-4} + 3.2 \times 10^{-7}(T - 773) \text{ [gr/cms]}\)

for N\(_2\): \(\mu = 3.9 \times 10^{-4} + (T - 773) \times 3.2 \times 10^{-7} \text{ [gr/cms]}\)

for Ar: \(\mu = 4.5 \times 10^{-4} + (T - 773) \times 4 \times 10^{-7} \text{ [gr/cms]}\)

when \(T\) = temperature in \(^\circ\text{K}\)

**Thermal Conductivity** (from ref [2.11])

for He: \(\lambda = 1.861 \times 10^{-4} + 5.912 \times 10^{-7} \times T \text{ cal/cms}^\circ\text{K}\)

for O\(_2\): \(\lambda = 5.861 \times 10^{-5}(T/273)^{0.87} \text{ cal/cms}^\circ\text{K}\)

for N\(_2\): \(\lambda = 5.77 \times 10^{-5}(T/273)^{0.8} \text{ cal/cms}^\circ\text{K}\)

for Ar: \(\lambda = 9.027 \times 10^{-5} + 9.25 \times 10^{-8}(T-773) \text{ cal/cms}^\circ\text{K}\)

when \(T\) = temperature \[^\circ\text{K}\]
B) Properties for the mixture

In order to calculate the properties of the environment as a function of its composition, the semi-empirical formula of Wilke (from ref. [2.12] was employed.

\[ \mu_{\text{mix}} = \sum_{i=1}^{n} \frac{X_i \mu_i}{\sum_{j=1}^{n} X_j \phi_{ij}} \]

in which

\[ \phi_{ij} = \frac{1}{\sqrt{8}} \left( 1 + \frac{M_i}{M_j} \right)^{-1/2} \left[ 1 + \left( \frac{\mu_i}{\mu_j} \right)^{1/2} \left( \frac{M_j}{M_i} \right)^{1/4} \right] \]

\( n \): number of chemical species in the mixture; for these calculations, \( n = 2 \)

\( X_i \) and \( X_j \): mole fractions of species \( i \) and \( j \)

\( M_i \) and \( M_j \): molecular weight of \( i \) and \( j \)

and

\[ \lambda_{\text{mix}} = \sum_{i=1}^{n} \frac{X_i \lambda_i}{\sum_{j=1}^{n} X_j \phi_{ij}} \]

in which \( \phi_{ij} \) is the same as above.
The purpose of this calculation is to illustrate the applicability of Helium to extinguish smoldering fires, and its comparison with other methods for one specific case. It is a rough estimate of the amount of extinguishant necessary to suppress a smoldering fire in the interior of a Boeing 707. This type of aircraft was selected for this calculation because several of the reported aircraft cabin fire [1.3] occurred on aircraft of this kind, and because it is a widely used type of aircraft, also because its dimensions were available in reference [4.13].

It is assumed that the initial environment in the airplane is air at 1 atm of pressure; from ref [4.13].

Volume of cabin = 7983 ft$^3$
Volume of baggage compartment = 1775 ft$^3$
Total interior volume = 9758 ft$^3$

Using as criterion for extinguishment that the oxygen mole fraction must be $X_{O_2} \approx 15\%$ [4.12]. It is determined that 11 bottles of Helium (type 1H of [4.6]) are needed to obtain those conditions, and the final pressure of the interior environment of the airplane would be of 1.4 atm. The cost of the helium (not including the bottles) would be about $495.00$ [4.6] and the weight of these 11 bottles is 2079 lbs.
The same calculation is performed for the case of $N_2$ in which the cost would be 28.5% lower and the bottles of $N_2$ are 11.11% heavier. Then even though $N_2$ is less expensive, its weight is, in this case, a disadvantage in front of Helium.

The extinguishant normally used in aircraft is "Halon 1301". The recommended concentration of Halon 1301 in the environment in order to extinguish a fire is [4.6] about 5 to 6%, which is lower than the necessary concentration of helium. However the Halon 1301 molecule is much heavier than the helium molecule (148.9 vs. 4) so the time that Halon 1301 needs to diffuse in the environment is also larger than the time the helium needs. For the case considered above the cost of the Halon 1301 needed to establish the extinguishment conditions is about 4 times that of Helium, which gives helium another advantage in this particular case.
FIGURE 2.1 Theoretical Model. Interactions between char-oxidation zone and pyrolysis zone, through the interface, and interactions with the environment.
FIG. 2.2  Physical Description of the Stationary Pyrolysis Zone. External Heat Flux ($q_s$) is imposed at $x = 0$. Virgin Material is entering zone and Char and Volatiles are leaving it.
FIG. 2.3 Pyrolysis Analysis for a Semi-Infinite Slab of Material

10, Comparison Between Numerical and Approximate Solution
FIGURE 2.4 Comparison between theoretical and experimental relationship between temperature and speed. Approximate and exact theoretical relationship included.
FIG. 2.5 Smoldering Combustion of a Sample of Cotton in a Known Atmosphere.
FIGURE 2.6 Heat generation and heat available curves for two samples of different diameter in air at 300°K and 1 atm.
FIGURE 2.7 Theoretical Temperature-Speed relationship for cellulosic materials with different densities. Solid lines are approximate solutions, the dashed line is the exact solution for $\rho = 0.06 \text{ gr/cm}^3$ (material #10).
FIGURE 2.8 Heat required for the pyrolysis process of materials with different densities, using approximate solution, equation 2.12
FIGURE 2.9  Heat generation \((q_g)\) and heat available \((q_a)\) curves as a function of temperature for different environmental conditions in He-O\(_2\). Material#10 (table II).
FIGURE 2.10  Graphical calculation of smoldering temperature.  
Comparison between approximate and exact solution for the pyrolysis study.  Material #10, smoldering in He-O\(_2\) 
\(X_{O_2}=40\%\), oxygen partial pressure \(P_{O_2}=12.5\) cmHg.
FIGURE 2.11 Comparison between the theoretical effect of oxygen mole fraction on the steady smoldering speed, for different inert diluents (indicated in each curve). For each curve the oxygen partial pressure is: In $N_2$, $P_{O_2}=15.5$ cmHg, in He $P_{O_2}=12.4$ cmHg, and in Ar $P_{O_2}=9.5$ cmHg. All calculations for material # 10.
FIGURE 2.12  Theoretical effect of partial pressure of oxygen on the smoldering speed for material #10. Comparison between the exact and approximate solutions, and between the effect in different environments.
FIGURE 2.13 Calculation of smoldering temperature for samples of different diameter and density smoldering in air at 1 atm of pressure.
FIGURE 3.1 Schematic diagram of pressure vessel
FIGURE 3.2 Metallic frame with four samples, as used in the experiments.
FIGURE 3.3 Radiation heat source with sample as mounted for experiments.
FIGURE 3.4 Schematic diagram of the load cell
FIGURE 3.5 Physical description of the smoldering sample as it is observed in the experiments.
FIGURE 3.5a  Sample of material #9 (Table II) smoldering in air at atmospheric conditions
FIGURE 3.6 Measured temperatures at different positions in a smoldering sample of material #10 (table II) in air at atmospheric conditions.
FIGURE 3.7 Density variation through the smoldering zone of a sample of material #10 smoldering in air at atmospheric conditions.
FIGURE 3.8 Comparison between experimental and theoretical effect of oxygen partial pressure on smoldering speed for material #10 smoldering in \( \text{N}_2-\text{O}_2 \) environments. Theoretical curves (solid lines) are for \( \chi_{\text{O}_2} = 21\% \).
FIGURE 3.9 Comparison between theory and experiments for the effect of oxygen partial pressure on the smoldering speed of material # 10, in He-O\textsubscript{2} environments. Dashed symbols correspond to experiments in N\textsubscript{2}-O\textsubscript{2} environments, included in this figure for comparison of this effect in different inert diluents.
FIGURE 3.10 Experimental dependence of smoldering zone temperature on oxygen partial pressure, in N$_2$-O$_2$ environments ($\chi_{O_2}=21\%$). Samples of material #10.
FIGURE 3.11 Effect of oxygen partial pressure on the temperature profile in the smoldering zone of samples of material #10. Environment at $X_2=21\%$ (air)
FIGURE 3.12 Comparison between theory and experiments for the effect of oxygen mole fraction on smoldering speed in Ar-O₂.
Dashed symbols represent experimental results with other inert diluents. In each case the oxygen partial pressure is:

For Ar-O₂ (○)  \( P_{O_2} = 9.5 \) cmHg

For \( N_2-O_2 \) (○)  \( P_{O_2} = 10.5 \) cmHg

For \( He-O_2 \) (□)  \( P_{O_2} = 9.9 \) cmHg
FIGURE 3.13 Comparison between experimental and theoretical dependence of smoldering speed on oxygen mole fraction. Material #10 smoldering in $N_2-O_2$ environments. Theoretical curves are for $P_{O_2} = 15.5$ cmHg.
FIGURE 3.14 Experimental and theoretical effect of oxygen mole fraction on the smoldering speed of material# 10 in He-O\(_2\) environments. The solid lines are the calculations for \( P_{O_2} = 12.4 \) cmHg.
FIGURE 3.15 Effect of the oxygen mole fraction on the temperature profile across the smoldering element. Samples of material #10 in N₂-O₂ environments.
FIGURE 3.16 Temperature of smoldering zone at different oxygen mole fractions in N₂-O₂ environments. Samples of material # 10 (table II).
FIGURE 3.17 Sample of a measurement of the temperature history inside a sample of cotton (material #10). For this particular case the environmental conditions are: $X_{O_2} = 0.50$, $X_{N_2} = 0.373$ and $X_{He} = 0.327$ and $P_{Total} = 37.5$ cm Hg.
FIGURE 3.18 Speed and temperature dependence on $\rho d$ for materials listed in table II. Solid lines represent theoretical calculations from table III. The theoretical curves indicate only the speed calculated. For experimental points (○) indicates the speed and (△) indicates the temperature. The experiments and the calculations are in air at atmospheric conditions.
FIGURE 3.19 Comparison between experimental and theoretical effect of density and diameter on smoldering speed. The environment in the tests and for the calculations, is air at atmospheric conditions (1 atm and 300°K)
FIGURE 3.20 Theoretical and experimental effect of diameter and density on the smoldering temperature. Experiments conducted in ambient air at atmospheric conditions (samples in table II). Solid lines are the calculations for: curve (1) d=0.14 cm, curve (2) d=0.22 cm, and curve (3) d=0.38 cm, also in air at atmospheric conditions.

Dashed symbols are indicating the temperature at the surface of the sample.

Diameters tested:
- 0.04 cms
- 0.13 "
- 0.17 "
- 0.375 "
- 0.6 "
- 0.86 "

[Graph showing temperature vs. density for different diameters.]
FIGURE 3.21 Effect of oxygen partial pressure on the steady smoldering speed on samples of different materials. Experiments in \( \text{N}_2-\text{O}_2 \) environments at \( \% \text{O}_2 = 21\% \) (air).
FIGURE 3.22 Smoldering regimes in $N_2-O_2$ material #10.
FIGURE 3.23 Smoldering regimes for samples of material #10 (table II) in He-O₂ environments. Solid lines are the calculated limits of extinguishment for different frequency factors (fₑ). An experimental point in Ar-O₂ environment is included for comparison.
FIGURE 3.24 Description of how the calculations are affected by the assumptions made. Heat losses effect in pyrolysis analysis: Figures a, b and c. Frequency factor effect: Figure d.
FIGURE 3.25 Distance-time (from ignition) curves, measured to determine the effect of the heating rate of initiation on the steady smoldering behavior. Experiments conducted in samples of material #10, in air at atmospheric conditions. For each curve the ignition source and initiation heat flux are:

Curve (1): Radiant heat source and 0.8 cal/cm².s
Curve (2): Radiant heat source and 1.  "
Curve (3): Radiant heat source and 1.3  "
Curve (4): Heating Coil and 1.  "

In all cases the ignition source is turned off at the observation of smoke.
FIGURE 3.26 Effect of heating rate of initiation on the ignition time. Comparison between the behavior of cotton and polyurethane. Experiments conducted in air at atmospheric conditions, using the radiant heat source as the ignition source. Samples of polyurethane were not able to sustain smoldering after the lamp was turned off.
FIGURE 4.1  Schematic description of experiment to test the effectiveness of helium as extinguishant agent.