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DEVELOPMENT AND FORMULATION OF COOLANT AND NON-CORROSIVE AEROSOL FORMING AGENT SYSTEM

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By

ZHANG XIAOTIAN

Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the Degree of Doctor of Philosophy

DEVELOPMENT AND FORMULATION OF COOLANT AND NON-CORROSIVE AEROSOL FORMING AGENT SYSTEM

By

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November 2015

Chair : Associated Professor Mohd Halim Shah Bin Ismail, PhD
Faculty : Engineering

A newly synthesized non-corrosive and effective aerosol fire extinguishing agent together with an effective coolant for hot aerosol cooling were successfully prepared and their properties and working performances were studied and discussed. The new S-type aerosol forming agent which contains 12.2% potassium nitrate, 44.2% strontium nitrate, 8.0% magnesium powder, 17.5% alpha-lactose, 11.1% ammonium nitrate and 7% epoxy binder was formulated to replace the corrosive K-type aerosol forming agents that currently dominates the aerosol fire extinguishant market. The fire suppression time, combustion reaction, combustion velocity, aerosol phase composition, combustion residue composition, aging profile and corrosion performance of this newly synthesized S-type aerosol forming agent were studied and analyzed. This new S-type aerosol forming agent could extinguish a heptanes pan fire in less than 20s in a 1 m^3 compartment and the discharged aerosol particles have surface resistivity larger than 20 MΩ which is the threshold insulation level for protecting the electrical devices and electronics according to the Aerosol Fire Extinguishing System, Part1: Condensed Aerosol Fire Extinguishing Device GA 499.1-2004 and GA 499.1-2010. Moreover, this is the first time in the research field of aerosol fire extinguishing agents that dynamic modeling of combustion temperature and pressure inside an aerosol forming agent canister were successfully achieved. During combustion, the temperature at the canister nozzle would rise to over 1000°C very quickly, thus a substantial cooling of the discharged aerosol using any coolant is compulsory. The simulation of the canister's inner pressure and temperature distribution on the coolant geometry provides instructive information on coolant geometry design and packing pattern of coolant. A packing bed randomly packed with Φ 5 mm kaolinite-based spherical coolants was used to cool down the hot aerosol and its temperature was brought down from 1400°C to below 400°C without any additional coolant. The mass of coolants applied is equal to the mass of the newly synthesized aerosol forming agent and the performance of such kaolinite-based coolant is relatively better than other common coolants used for hot aerosol cooling. Properties such as yield stress, thermal conductivity, water activity as well as manufacturing process of kaolinite-based spherical granulate were studied and discussed. The composition for synthesizing the kaolinite-based coolant used in this study is as follows: 42.8% kaolinite powder, 38.8% water, 8.1% epoxy A&B glue, 10.2% ethyl cellulose, and the evaporation of the absorbed water in the kaolinite mineral plays a major role in hot aerosol cooling.

Abstrak tesis yang dikemukakan Kepada Senat, Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

PEMBANGUNAN DAN FORMULASI SISTEM PENDINGIN DAN PEMBENTUKAN AEROSOL TIDAK HAKIS

By

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Satu sistem terdiri daripada agen aerosol baharu yang tidak menghakis dan berkesan untuk pemadaman api, beserta satu bahan pendingin yang berkesan. Telah berjaya disediakan. Sifat-sifat serta prestasi kerja kedua-duanya telah dikaji dan dibincangkan. Agen pembentuk aerosol jenis-S yang baharu ini mengandungi 12.2% kalium nitrat, 44.2% strontium nitrat, 8.0% serbuk magnesium, 17.5% laktosa-alpha, 11.1% ammonium nitrat dan 7% pengikat epoksi dicipta untuk menggantikan agen pembentuk aerosol jenis-K yang bersifat menghakis yang mendominasi pasaran pemadam api aerosol masa kini. Masa untuk memadamkan api, tindak balas pembakaran, halaju pembakaran, komposisi fasa aerosol, komposisi sisa pembakaran, profil penuaan, dan prestasi pengaratan bagi agen pembentuk aerosol jenis-S yang baru disintesis ini telah dikaji dan dianalisis. Agen pembentuk aerosol jenis-S baharu ini mampu memadamkan api di dalam bekas berisi heptana kurang daripada 20 saat bagi ruang 1 m^3 dan zarah-zarah aerosol yang dibebaskan mempunyai rintangan permukaan melebihi 20 MΩ, iaitu tahap ambang penebat bagi melindungi alatan elektrik dan elektronik menurut Sistem Pemadam Api Aerosol, Bahagian 1: Alatan Pemadam Api Aerosol Memeluwap GA 499,1-2004 dan GA 499,1-2010. Sebagai tambahan, buat kali pertamanya di dalam bidang penyelidikan agen pemadam api aerosol, satu pemodelan dinamik tentang suhu dan tekanan pembakaran di dalam tabung agen pembentuk aerosol telah berjaya diperolehi. Semasa kebakaran, suhu pada muncung tabung boleh meningkat kepada 1000°C atau lebih dengan cepat, maka suhu aerosol yang dilepaskan mesti disejukkan secukupnya dengan menggunakan bahan pendingin. Simulasi tentang tekanan dalaman dan agihan suhu di dalam tabung terhadap geometri bahan pendingin telah memberikan maklumat penting untuk reka bentuk geometri dan pola pemadatan bahan pendingin. Satu turus terpadat yang diisi secara rawak dengan bahan pendingin berasaskan kaolinit, berbentuk sfera dengan diameter 5mm, telah digunakan untuk menyejukkan aerosol panas dan suhunya berjaya diturunkan daripada 1400°C kepada kurang 400°C tanpa menggunakan sebarang bahan pendingin tambahan. Jisim bahan pendingin yang digunakan adalah sama dengan jisim agen pembentuk aerosol yang baru disintesis tersebut dan prestasi bahan pendingin berasaskan kaolinit ini adalah lebih baik daripada bahan-bahan pendingin lain yang biasa digunakan untuk menyejukkan aerosol panas. Sifat-sifat seperti tegasan alah, konduktiviti termal, aktiviti air dan juga proses pembuatan butiran sfera berasaskan kaolinit telah dikaji dan dibincangkan. Komposisi penyediaan bahan pendingin berasakan kaolinit yang digunakan di dalam kajian ini

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adalah seperti berikut: 42.8% serbuk kaolinit, 38.8% air, 8.1% pelekat epoksi A & B, 10.2% etil selulosa dan penyejatan air yang terserap pada mineral kaolinit memainkan peranan penting dalam penyejukan aerosol panas.

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This thesis was submitted to the Senate of Univerisiti Putra Malaysia and has been accepted as fulfilment of the requirement for the Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AFA	aerosol forming agent
ALT	Atmospheric Lifetime
DSC	Differential Scanning Calorimetry
G1	Generation 1
G2	Generation 2
G3	Generation 3
GWP	Global Warming Potential
LOAEL	Lowest Observed Adverse Effect
Max	Maximum
ODE	Ordinary Differential Equation
ODP	Ozone Depletion Potential
PCCs	Phosphorus Containing Compounds
PDE	Partial Differential Equation
PVC	Poly Vinyl Chloride
R&D	Research and Development
Redox	Reduction-oxidation
SEM-EDX	Scanning Electron Microscopy with Energy Dispersive X-ray spectroscopy
STD	standard deviation

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LIST OF NOMENCLATURES

Symbol	Definition	SI Unit
A	Intersection area of tank	m^2
A_N	Cross section of canister	m^2
A_{in}	Inner surface area of canister	m^2
a_v	Volume function constant	m^3/s^2
b	Canister wall thickness	m
b_v	Volume function constant	m^3/s
c	Voids of packing bed	m^3
C_2	Designed fire extinguishing concentration	kg/m^3
C_{pc}	Steel canister heat capacity	J/g•K
Cp_i	Heat capacity of each reactant and product	J/g•K
$\overline{C}_{pg}$	Average heat capacity of aerosol gases	J/g•K
$\overline{C}_{ps}$	Average heat capacity of aerosol particles	J/g•K
d_p	Diameter of primary particles	m
D	Diffusion coefficient	m^2/s
D_f	Fractal dimension	?
D_p	Equivalent spherical diameter of the packing granulate	m
g	Gravitational acceleration	m/s^2
i	Emissions source for primary particles	
j	Particle size	m
K	Compensation coefficient	?
k_f	Fractal prefactor	?
K_v	Volume correction coefficient	?
K_{TC}	Total heat transfer and conduction coefficient	W/m^2•K
L	Packing bed length	m
L_a	Height of AFA block	m
L_c	Length of canister cylinder	m
m	Number of particle of different sizes	?
M	Dosage	kg

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M_c	Canister mass	kg
$\overline{M}_g$	Average gases molar mass	g/mol
n	Number of primary particles	-
N	Number density	m^{-3}
N_A	Avogadro number	mol^{-1}
P_o	Atmospheric pressure	Pa
$Q_{Accumlated}$	Accumulated heat	J/s
Q_e	Extinction coefficient	-
Q_s	Scattering coefficient	-
Q_{in}	Source heat generated	J/s
Q_{out}	Heat flux leaving canister	J/s
$Q_{Phase\ change}$	Phase change heat	J/s
Q_r	Radiation heat	J/s
Q_{TC}	Transfer and convection heat	J/s
r_i	Inner radius of canister cylinder	m
r	Numerical radius of particles	m
R	Ideal gas constant	J/mol•K
RAB	Mass ratio of Epoxy A&B binder	-
Re	Effective forming agent dosage per storage area of tank	kg/m^2
R_g	Radius of gyration of an aggregate	m
RK	Mass ratio of kaolinite	-
RKW	Mass ratio of kaolinite/ water	-
Rw	Mass ratio of water	-
T_0	Initial coolant body temperature	K
$T_{Ae}(t)/T$	Temperature of hot aerosol	K
T_{Air}	Atmospheric temperature	K
T_B	Temperature on outer surface of perforated coolant cylindrical	K
T_I	Redox temperature of AFA block	K
T_H	Inner surface temperature of canister wall	K
T_L	Outer surface temperature of canister wall	K
T_{ND}	Aerosol temperature at nozzle	K

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V	Volume under protection	m^3
V_c	Gaseous volumetric flow	m^3/s
V_e	Aerosol velocity passing through an empty canister	m/s
V_s	Average velocity of gases leaving the canister at the nozzle	m/s
W	Burning rate of AFA	kg/s
Wa	Kaolinite based clay water activity	-
W_d	Designed dosage	kg
X	Molar ratio	□
Y	Kaolinite based clay yield stress	kPa
α_c	Inversed heat diffusivity of coolant material	s/m^2
α_i	Convection heat transfer coefficient	$W/m^2 \bullet K$
α_g	Gas adsorbance	□
α_o	Natural convection heat transfer coefficient	$W/m^2 \bullet K$
δ	Thickness of perforated cylinder wall	m
ε	Packing porosity	-
η	Dynamic viscosity of disperse medium	$Pa \bullet s$
λ_m	Conductivity of canister wall	$W/m \bullet K$
μ_g	Aerosol gases dynamic viscosity	$Pa \bullet s$
ν	Terminal velocity of spherical particles	m/s
ε_g	Gas emmittance	□
ε_m	Canister wall emmittance	□
ρ	Density of particles	kg/m^3
ρ_0	Density of disperse medium	kg/m^3
ρ_g	Density of aerosol gases	kg/m^3
σ	Blackbody radiation constant	$W/m^2 \bullet K^4$
υ_i	Stoichiometric number	□

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CHAPTER 1

INTRODUCTION

1.1 Background

Fire extinguishing technology has long been developed in modern human history, and so far it can be classified into inert gas, CO_2, halogen agent, foam, dry powder, water mist or stream and condensed aerosol fire suppression systems. For inert gas and CO_2 fire suppression system, it uses pre-sorted high pressure inert gas or CO_2 for fire extinguishing by cooling and choking of fires through local or total flooding application, but the quantity needed is high and inert gas or CO_2 can cause choking or freezing hazards to humans when they are applied during fire suppression. Halogen fire extinguishing systems like Halon fire suppression systems were once very popular in the market due to its high fire suppression efficiency, but as the problem of ozone depletion caused by using Halon extinguishing agents which contain bromofluoroalkanes (Andrzej & Tsang, 1997), many countries joined the Montreal Protocol which announced the protective actions for ozone layer in 1987, and such act promoted the phase out of Halon extinguishants. Foam fire suppression system is quite efficient for Class B fire (combustible liquid) extinguishing and it forms a foam layer on the combusting liquid and chokes the fire, but such fire suppression system requires large amount of foams and the foams are hard to clean. Dry powder fire suppression system (e.g. ABC powder fire suppression system) has the caking and deliquescence problem of powders and the powders for fire suppression is dirty and corrosive to the environment. Also it is hard to apply dry powder fire suppression system in a total flooding fire suppression mode. Water stream fire extinguishing is clean and cheap, but it has low fire extinguishing efficiency, and water mist fire extinguishing system requires highly pressurized delivery system for a total flooding fire suppression mode.

With the development of fire science and fire technology, new aerosol fire suppression system which includes pre-dispersed particles (cold aerosol) fire suppression and condensed/pyrotechnic aerosol (hot aerosol) fire extinguishing system has been widely applied nowadays. The fire extinguishing mechanism behind condensed aerosol fire suppression is combustion radicals binding and choking. Especially in recent years, hot aerosol fire extinguishing technology has attracted more and more attentions, and such fire extinguishing system is highly efficient compared to traditional fire suppression systems like inert gas, CO_2 and water stream systems. In Malaysia, the market for hot aerosol fire extinguishing system is still growing, some local companies and manufacturers like "Pyrogen Manufacturing Sdn Bhd" has become one of the leader in local hot aerosol fire suppression system manufacturing and distribution. So it is interesting and important to conduct a research on hot aerosol fire extinguishing technology to help better promote the application of such technology in Malaysia.

Hot aerosol fire extinguishing technology was first proposed in 1960s based on pyrotechnics technology in rocket science and it continues developing since then. Hot

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aerosol extinguishing agents have high efficiency in fire extinguishing and they do not need to be stored in a pressurized container since they do not need to be driven out by pressurized inert gases. A hard block of hot aerosol fire extinguishing agent sits in an extinguisher canister which has a perforated cap called nozzle, and a fuse attached to the canister ignites the aerosol forming agent to generate the fire extinguishing aerosol which is carried out by aerosol gases produced.

Different from traditional or cold aerosol extinguishing agents, during combustion of hot aerosol fire extinguishing agent, hot aerosol colloids with diameters from 10^{-9} to 10^{-7} meters are generated by redox (reducing-oxidizing) reaction of components in hot aerosol forming agent. Those colloids have diameters much smaller than 4×10^{-6} m at which Brownian motion starts to occur (Fu et al., 2001) and those tiny aerosol particles have high diffusivity and long time to suspend in a fire zone for fire fighting. Generally, the mechanisms behind fire suppression by hot aerosol fire extinguishing agents are both physical and chemical. Physical mechanisms include oxygen isolation and temperature cooling, while chemical mechanism is recombination of fire supporting radicals.

Aerosol forming agent block can be stored under the atmospheric pressure and have low Ozone Depletion Potential (ODP) and Global Warming Potential (GWP) values (Guo & Yue, 2008). Through 40 years development of hot aerosol fire suppression technology, it has come to the third generation of aerosol fire suppression agent which is called S-type (G3) hot aerosol extinguishing agent using strontium nitrate as main oxidant in the aerosol forming agent. But currently, traditional K-type (G2) aerosol fire extinguishing agent which uses potassium nitrate as major oxidant in its hot aerosol forming agent is still dominant in the market of hot aerosol fire extinguishing agents.

1.2 Problem statement and hypothesis

Hot aerosol fire suppression system is a new fire suppression system which is not well developed compared to inert gas, CO_2, halogen agent, foam and water mist or stream fire suppression system, so is the fire suppression agent used in hot aerosol fire suppression system. Aerosol particles generated by K-type product are corrosive to electrical devices and electronics, thus it is very necessary to develop a non-corrosive hot aerosol fire suppression agent. Also this research purposes to analyze the physical and chemical properties of the novel hot aerosol forming agent synthesized and aims to model and calculate the aerosol temperature and pressure dynamics during aerosol forming agent combustion inside the canister since little knowledge is known about the combustion process of hot aerosol forming agent in a canister. Last, high temperature of aerosol discharged during aerosol forming agent combustion is still a problem which limits the application of hot aerosol fire suppression system, so this research will design and synthesize a new coolant system for hot aerosol cooling.

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The current research also hypothesizes that:

1. Corrosion problem caused by K-type hot aerosol forming agent can be largely alleviated by applying a new S-type hot aerosol forming agent.

2. Models can be established to predict the canister nozzle temperature and understand the inner canister pressure during new S-type aerosol forming agent combustion.

3. A cooling bed composed of novel synthesized coolants can efficiently reduce the temperature of hot aerosol compared to some traditionally used coolants.

1.3 Importance of study

This research deals with the synthesis and property analysis of a novel non-corrosive hot aerosol forming agent which can be applied in occasions where delicate electronics and electrical equipments or important archives are presented. Also the research will synthesize efficient coolants for hot aerosol cooling and a hot aerosol fire suppression system which adopts the new non-corrosive hot aerosol forming agent and the new coolants can be widely used and capable to replace the current corrosive K-type hot aerosol fire suppression systems.

Moreover, this research will establish models to describe dynamic changes of nozzle temperature and inner canister pressure during novel hot aerosol forming agent combustion for the design of an efficient coolant system and it helps to increase our knowledge and understanding of the combustion process of hot aerosol forming agent.

1.4 Objectives

The general objective is to develop a new S-type fire suppressive hot aerosol forming agent which generates non-corrosive aerosol to most electronics and electrical devices, and understand the new S-type aerosol combustion process to develop a coolant part for the hot aerosol cooling. The specific objectives are following:

1. To formulation and optimization of a novel non-corrosive aerosol forming agent which is effective in fire extinguishing. The corrosiveness and fire suppression efficiency of the novel non-corrosive aerosol forming agent should meet the requirements in Standards GA 499.1-2010.

2. To synthesize new coolants which are kaolinte based and design the coolant packing pattern for the hot aerosol fire suppression agent. The cooling efficiency of the new coolant system should be high and stable compared to other commonly used coolants for hot aerosol cooling.

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3. To model the dynamic changes of nozzle temperature and inner canister pressure based on redox/combustion process study of the novel non-corrosive hot aerosol forming agent.

4. To compare the predicted nozzle temperature with experimental data.

1.5 Scope and limitation of study

The current research only focuses on the synthesis of a new non-corrosive aerosol forming agent, nozzle temperature and inner canister pressure modeling and synthesis of a new type of coolant and its packing design for the hot aerosol cooling. The research will not deal with the design and installation of the hot aerosol fire extinguishing canister/extinguisher, nozzle and ignition system. The whole hot aerosol fire suppression system is a comprehensive and complicated system, and it is impossible to cover all the aspects of hot aerosol fire suppression technology in the current research. Currently, the application of hot aerosol fire suppression system is limited to countries as USA, Russia and China, etc, and it has not been applicable in Malaysia.

1.6 Thesis layout

The thesis is composed of five Chapters. Chapter 1 is "introduction" which includes background, problem statement and hypothesis, importance of study, objectives, scope and limitations of study and thesis layout. Chapter 2 is "literature review" which describes the develop history, fire suppression mechanism, chemical properties and applications of fire suppressive hot aerosol forming agent and the types, properties and applications of hot aerosol coolants. Chapter 3 is "materials and methodology" regarding the formation of research objectives, experimental designs, data collection and analyses. Chapter 4 is "results and discussions" which explains the data and phenomena collected and observed from the experiments and modeling. Last, Chapter 5 is "summary and recommendation" which concludes the finds and contributions of the study.

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