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Ferrocene-containing compounds as combustion catalysts and solid fuel modifiers

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Abstract. Low pressure profile and stable burning rate are important and necessary characteristics of high-quality solid fuels. Achieving their optimal values is accomplished by incorporating combustion catalysts. There are several types of combustion catalysts, such as metal nanoparticles, oxides, transition metal chelates, and catalytic mixtures based on them. Among catalysts, ferrocene and its compounds hold a special place. They are widely used in the aerospace industry due to their superior microscopic homogeneity, proper ignitability of rocket fuel, and compatibility with organic binders. However, ferrocene compounds tend to migrate within the composite, leading to matrix degradation, reduced storage life, and shorter operational lifespan of the fuels. Polymeric ferrocene catalysts represent a new generation of catalysts that retain activity while exhibiting reduced migration tendencies. They have a polymeric structure in which the ferrocene group can be placed in the main or side chain.

In this study, in addition to reviewing current knowledge on polymeric ferrocene combustion catalysts, synthesis methods and their application results were examined, as well as their migration in fuels compared to other catalysts. The conducted research demonstrated that polymeric ferrocene catalysts are synthesized through free-radical and graft polymerization, resulting in dendrimer-like polymers. Furthermore, the use of a hyperbranched polymeric ferrocene catalyst, compared to a ferrocene catalyst bound to a small molecular group, simultaneously reduced the migration rate by 90%. The iron content in the catalyst, the polymer's molecular weight, the placement of ferrocene in the polymer structure, and the degree of linearity of the polymeric structure are among the most important factors influencing the efficiency of these catalysts.

Keywords: ferrocene, ferrocene combustion catalyst, composite solid fuel, migration reduction, solid fuel burning rate

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1. INTRODUCTION

To overcome earth's gravity and launch satellites, rockets and other payloads into orbit, propulsion systems are often employed. The solid fuels used for this purpose are characterized by a low pressure profile and stable burning rate. A key method for increasing the burning rate of engines and eliminating primary smoke is the use of combustion catalysts in the fuel formulation [1].

Combustion catalysts (CCs) primarily include transition metal oxide nanoparticles, metal (organic) chelates, and catalysts based on ferrocene (Fc) and its derivatives [1, 4].

Metal nanoparticles with a high surface area possess elevated surface energy, which promotes an increased burning rate. Transition metal oxides, such as Fe_2O_3 or $CuOCr_2O_3$, are easily and inexpensively synthesized but lack the ability to significantly enhance the burning rate. Organic chelates of metals such as copper or iron with lead exhibit good dispersing properties and substantially increase the burning rate. Ferrocene-based catalysts are most commonly used in solid propellants due to their excellent ignition properties, uniform microscopic distribution, and superior compatibility with organic binders compared to other combustion catalysts [1, 2].

This paper presents the most common types of combustion catalysts and their classification. Subsequently, in line with the main objective of the article, polymeric and composite ferrocene-containing catalysts are examined. Ferrocene (Fc) – an organometallic compound with the formula $Fe(C_5H_5)_2$ – contains two cyclopentadienyl groups positioned on either side of the central iron atom and bonded to it (Fig. 1). Due to its unique aromatic structure, the applications of ferrocene derivatives are highly diverse, for example, they can be used as combustion catalysts [3, 5].

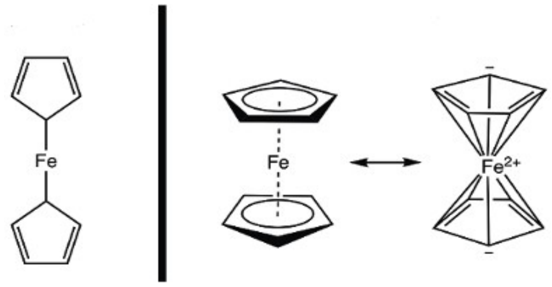


Fig. 1. Structure of ferrocene.

Following the accidental discovery of ferrocene (Fc) in 1951, numerous its derivatives were synthesized and identified. Ferrocene itself is a volatile red crystalline solid that has been used since its discovery as a catalyst for motor fuel oxidation. Ferrocene demonstrates higher efficiency compared to other metal compounds such as iron oxide, copper chromite, etc. It provides higher burning rates and can be effectively used at higher concentrations to achieve significant combustion rate enhancement. However, unfortunately, due to ferrocene's high volatility, the rocket propellant formulation changes over time, resulting in deterioration of mechanical and ballistic properties of the rocket propellant. Additionally, ferrocene promotes corrosion processes. Therefore, significant efforts have been directed toward developing new ferrocene compounds [6].

Based on chemical structure, ferrocene catalysts are divided into three categories depending on the attached organic moiety: a) alkylferrocenes, b) ferrocene oligomers, and c) ferrocene polymers. Among these, polymeric composite materials based on ferrocene and its derivatives have found wide application due to their significant impact on the burning rate of fuels containing ammonium perchlorate (AP) - the most common oxidizer in composite solid propellants [7, 8].

The effect of ferrocene-based polymers on burning rate depends on their chemical structure, solubility, molecular mobility, and amount of active iron. Ferrocene-based polymers are classified into main-chain polymers with (Fc) as a cyclic group in the backbone, branched ferrocene-containing polymers (hyperbranched) and their derivatives, which will be discussed later. The latter possess several unique properties such as low viscosity, high molecular weight, and excellent catalytic and electrical properties [9].

Ferrocene-based combustion catalysts are divided into five categories according to their application and role in propellant formulations: 1) ferrocene derivatives as plasticizers, 2) ferrocene derivatives as adhesives, 3) ferrocene derivatives as curing agents (in co-curing systems), 4) ferrocene derivatives as binding agents, 5) other ferrocene derivatives[10].

Interestingly in ferrocene derivatives of categories 1, 2 and 3 the migration problem is largely solved by incorporating ferrocene into the matrix chemical structure. For example, glycidyl ether of poly (bisphenol-A and 1,1-ferrocenedicarboxylic acid) (GEP-BFC) (Fig. 2) reacts through its epoxy group with the propellant curing agent, thus participating in the formation of three-dimensional cross-linked structures, which significantly reduces its migration rate [11, 12].

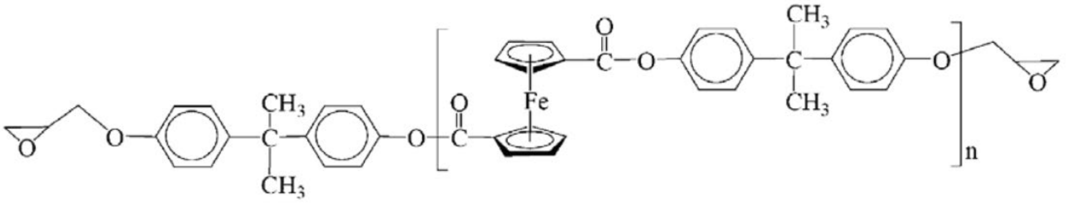


Fig. 2. Structure glycidyl ether of poly (bisphenyl-A-ferrocene-1, 1-dicarboxylate) (GEP-BFS).

2. METHODS AND MATERIALS

As the polymer binder, an industrial-grade hydroxyl-terminated polybutadiene (HTPB) resin with a molecular weight of 6400 ± 500 was used.

Benzoyl peroxide (BP) was employed as the initiator, which was recrystallized twice from methanol and dried under vacuum to constant weight at room temperature.

Ferrocene (Fc) of "chemically pure" grade was purified by sublimation before use. Vinylferrocene (VFC) was commercially obtained with a purity of 99.0% and a melting point of 51°C.

Zinc oxide was of "chemically pure" reactive grade. Ammonium perchlorate (AP) was of industrial "chemically pure" grade, with a main substance content of 99%.

Preparation Method of Compositions

A sample of 0.0465 g (2×10^{-4} mol) of benzoyl peroxide (BP) was dissolved in benzene, followed by the addition of 3 g HTPB and the calculated amount of vinylferrocene (Table 1). To homogenize the mixture, it was heated with stirring to 75°C [16]. The resulting solution was purged with gaseous nitrogen for 24 hours, then precipitated in methanol, washed several times with methanol, and dried under vacuum at room temperature. Several samples were prepared, and the compositions of the initial mixtures are presented in Table 1.

Table 1. Composition of the initial mixture for polymer catalyst synthesis.

№ Sample	Components of the Mixture (g)		
	Benzoyl Peroxide	Vinylferrocene	HTPB Resin
1	---	0.064	3
2	0.0465	0.064	3
3	0.0465	0.100	3

The obtained samples were characterized using Fourier-transform infrared (FT-IR) spectroscopy, thermogravimetric analysis (TGA), and gel permeation chromatography (GPC). A Perkin Elmer (USA) FT-IR-100 spectrometer was used for IR spectroscopy.

Gel permeation chromatography was performed on a Waters Alliance GPC/V 2000 instrument equipped with four columns packed with Waters Styragel (pore sizes 10^3–10^6 Å). The separation temperature was 30°C, with tetrahydrofuran as the eluent at a flow rate of 0.5 mL/min.

Thermogravimetric studies were conducted using an SDT Q600 instrument (TA Instruments, USA) with a heating rate of 0.5°C/min [13].

Synthesis of polymeric ferrocene-containing combustion catalysts

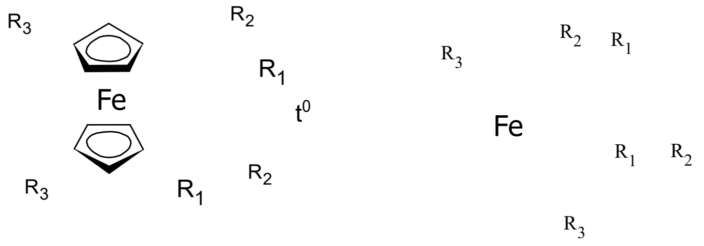
Synthesis of polymeric combustion catalysts containing ferrocene units in the main chain

Various methods for synthesizing polymers with catalytic properties based on ferrocene (Fc) in the main chain have been proposed, such as: ring-opening polymerization (ROP), metathesis polymerization, and cyclization reactions [14].

Synthesis by transition metal-catalyzed ring-opening polymerization

Transition metal-catalyzed ring-opening polymerization (ROP) is a convenient method for synthesizing high-molecular-weight metal-containing polymers. Using Pd and Pt catalysts, poly (ferrocenylsilanes) with high molecular weight were synthesized through the polymerization of silicon-bridged ferrocene (Fig. 3) via the substitution of chlorine and methyl groups at the silicon atom (Fig. 4).

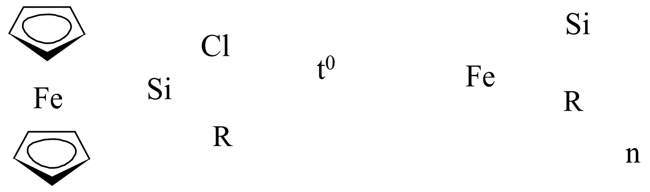
Since the angle between the two cyclopentadienyl rings in ferrocenyldialkylsilane with a silicon bridge is 20.8°**,** the molecule exhibits significant ring strain (~80 kJ/mol**)**, making it highly susceptible to ring-opening polymerization (Fig. 4) [15].



$R_1=R_2=R_3=H$

$R_1=R_2=H$ $R_3=Me$

Fig. 3. Thermal ring-opening polymerization reaction.



1. R=H
2.R=Me

Fig. 4. Pd-catalysed polymerization reaction of ferrocenyl chloroalkylsilane cycle opening.

Synthesis by anionic polymerization with cycle opening

The ring-opening thermal polymerization method is widely used to produce high molecular weight polymers, but the final products have a wide molecular weight distribution.

Ferrocene-containing monomer (Fig. 5, R= H) is used to obtain polymers with high molecular weight M_N and very narrow molecular weight distribution (polydispersity 1.05 - 1.10). The process is carried out in tetrahydrofuran (THF) solvent using $n-\mathrm{BuLi}$ or PhLi as initiator at 25 °C (Fig. 5).

It was found that anionic polymerization with cycle opening involves nucleophilic attack on the silicon atom to form an anion (initiator) and the subsequent participation of this anion in chain growth. It is important to note that the process is a living polymerization and therefore requires high-purity monomer and polymerisation medium [16, 17].

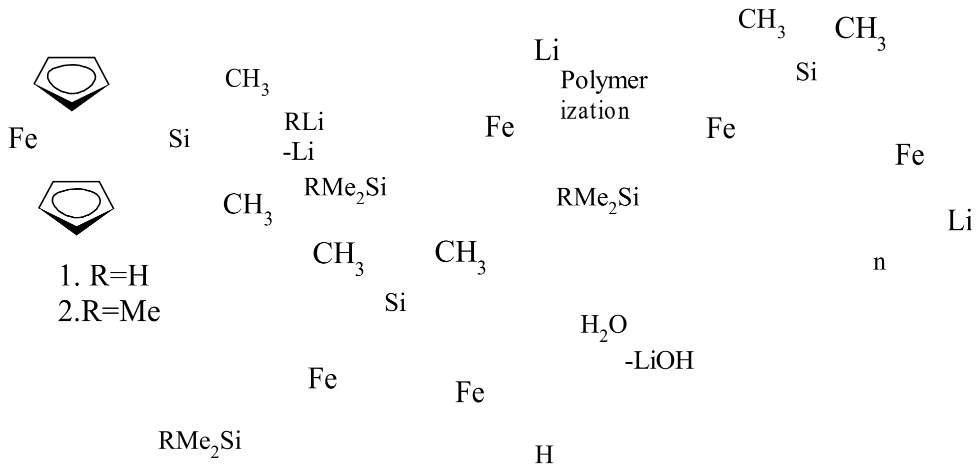


Fig. 5. Anionic ring-opening polymerization of silicon-containing ferrocene.

Synthesis of combustion catalyst polymers with ferrocene in side chains

Although methods for synthesizing polymers with ferrocene (Fc) in the *main chain* have been well-established over the past two decades, the synthesis of polymers incorporating ferrocenyl groups in *side chains* has gained attention only recently. This is due to the development of novel controlled radical polymerization techniques (e.g., complex-radical polymerization) in recent years. These methods enable the production of structurally diverse polymers with controlled molecular weight, narrow polydispersity and high functionality [18].

Free-radical polymerisation (FRP), cycloaddition (CA), atom transfer polymerisation radical addition and chain transfer fragmentation are common methods for the synthesis of polymeric ferrocene-based combustion catalysts containing ferrocene in the side chain [19].

Synthesis by free-radical polymerization

The synthesis of ferrocene-based polymers through free-radical polymerization (FRP) can be performed under various reaction conditions. However, the process parameters are highly sensitive to the purity of monomers and reagents. In this study, FRP was used to synthesize a polymer from vinylferrocene grafted onto methyl-β-cyclodextrin, as illustrated in (Fig. 6) [20, 21].

Synthesis by reaction

Electron donor-acceptor conjugated polymers find applications in organic photovoltaic devices with nonlinear optical system for semiconductor devices, etc. Therefore the polymer production reaction by copolymerisation provides a convenient method for the synthesis of donor-acceptor conjugated polymers. It has also been found that chemical doping increases the conductivity of ferrocene-based compounds, but its disadvantage is that it reduces the strength of the synthesised polymer. In addition, ferrocene exhibits effective electron-donor properties in the course of such a reaction [22, 23].

Synthesis via Copolymerization Reaction

Conjugated donor-acceptor polymers have found applications in organic photovoltaic devices with nonlinear optical systems semiconductor components and related technologies. The copolymerization approach thus provides a convenient synthetic route for producing donor-acceptor conjugated polymers.

Studies have demonstrated that chemical doping enhances the conductivity of ferrocene-based compounds, though this improvement comes at the expense of reduced polymer strength. Notably, ferrocene exhibits exceptional electron-donating properties in such reactions, making it particularly effective for these systems [24].

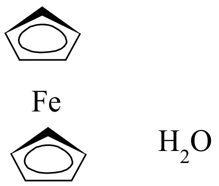


Fig. 6. Synthesis scheme for ferrocene-grafted methyl-β-cyclodextrin.

Copolymerization represents an effective method for synthesizing combustion catalysts containing ferrocene in side chains. For instance, ferrocenylacetylene can be incorporated into the conductive polymer poly (3, 4-ethylenedioxythiophene) by copper-catalyzed azide-alkyne cycloaddition (click chemistry), as illustrated in (Fig. 7) [25].

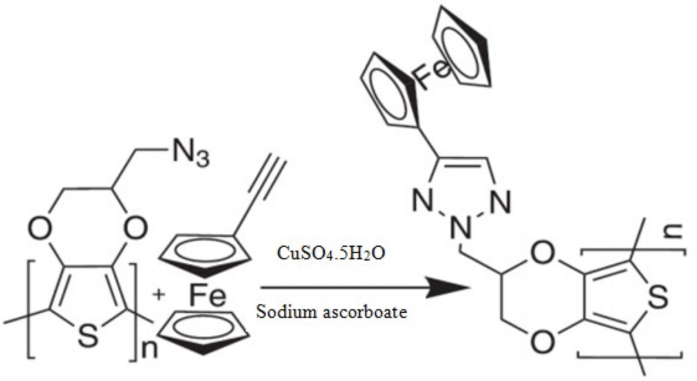


Fig. 7. Cycloaddition reaction of ferrocenylacetylene to the ring catalysed by Cu.

Synthesis via Atom Transfer Radical Polymerization

Controlled radical polymerization stands as the most promising method for producing dendrimeric polymers. This technique enables the synthesis of copolymers with precise compositional ratios and controlled molecular weights. The reaction can be conducted in aqueous media and by leveraging the reactivity of organic functional groups it becomes possible to create polymeric products with high, spatially differentiated density.

In this study the unique properties of poly(2-(methacryloyloxy)ethyl ferrocenecarboxylate) were utilized to synthesize a dendrimer on crosslinked polystyrene particles (Fig. 8) [26, 27].

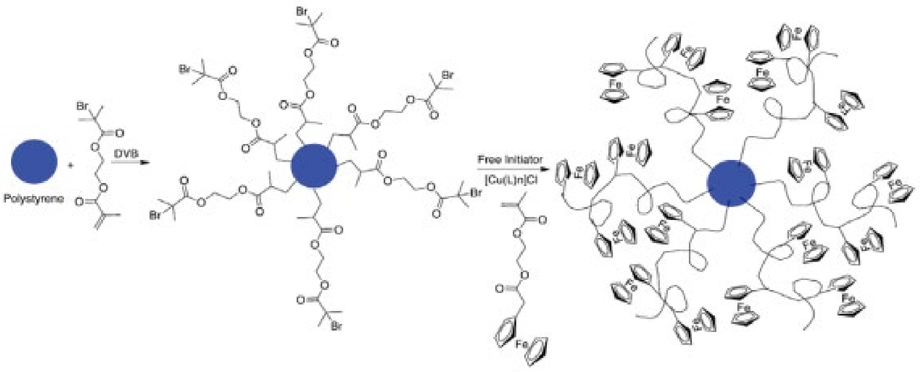


Fig. 8. Synthesis of a dendrimeric polymer incorporating 2-(methacryloyloxy)ethyl ferrocenecarboxylate.

Synthesis via Chain-Transfer Polymerization

The ferrocene-containing monomer ferrocen-1-yl-ethyl benzodithioate (FEB) was synthesized and employed as a chain-transfer agent in the polymerization of styrene. The polymerization pathway is illustrated in Fig. 9 [28].

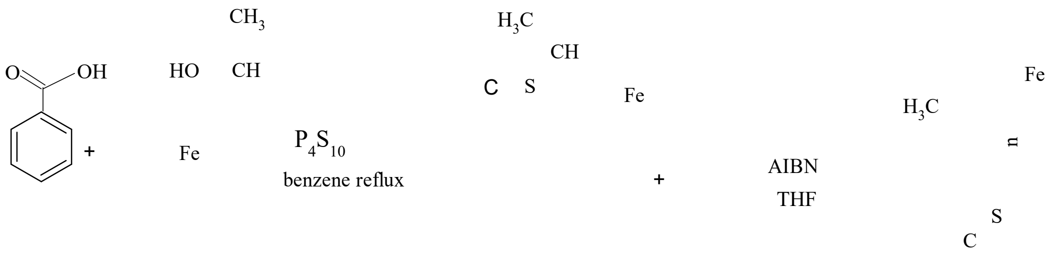


Fig. 9. Synthesis of a ferrocene-containing monomer and its role in styrene polymerization.

Synthesis of ferrocene derivatives as combustion catalysts

Ferrocene derivatives are widely employed as combustion catalysts, particularly in systems such as hydroxyl-terminated polybutadiene (HTPB). Among these, bimetallic ferrocenyl compounds demonstrate superior performance due to their excellent thermal stability, low volatility, and minimal matrix migration. The most significant ferrocene-based combustion catalysts include: 2, 2-Bis(ethylferrocenyl)propane (catocene, Fig. 10), its HTPB-grafted derivatives such as butacene (Figure 10), these compounds maintain low pressure profiles while preserving the essential mechanical properties of the fuel systems [29, 30].

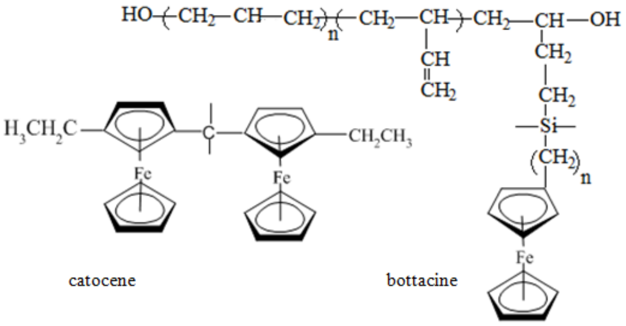


Fig. 10. Structure of butacene.

3. RESULTS AND DISCUSSION

Many methods have been proposed to reduce or prevent the migration of ferrocene compounds. Overall, there are two maintheoretical solutions to this problem: a) Increasing the alkyl chain length of substituents on the ferrocene rings, which increases the volume and mass of ferrocene-containing compounds, b) Formation of a cross-linked network structure: by adding active functional groups to ferrocene compounds to create a physical network and subsequently cross-linking it through a reaction with a curing agent (Fig. 16). In such a system, ferrocene molecules lose their ability to evaporate and can only undergo thermal vibrations with minimal movement within the composite.

Catalytic properties of polymeric ferrocene catalysts of combustion

In this study, the role of poly (ferrocenyldimethylsilanes) (PFDMS) and poly(ferrocenylphenylmethylsilanes) (PFDMS) with different molecular weights and polydispersity as combustion catalysts in the production of propellant containing AP/HTPB/hexamethylene diisocyanate. Fig. 11 shows the thermal decomposition data of pure AP (curve 1), propellant containing only ferrocene (outside the polymeric structure) (curve 2), and propellant containing polymeric catalyst PFDMS (curve 3).

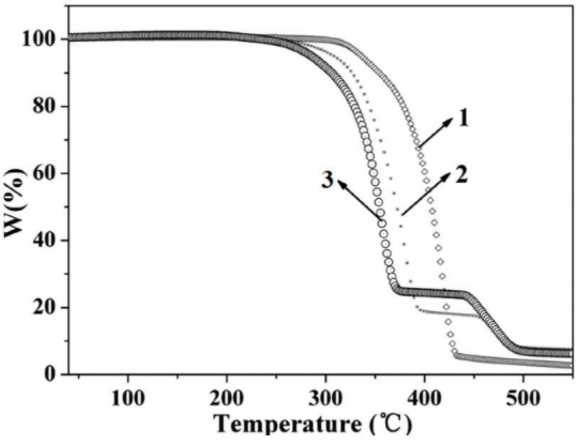


Fig. 11. TGA profiles for: (1) pure AP sample (curve 1); (2) propellant containing ferrocene (curve 2), (3) propellant containing PFDMS with number-average molecular weight (14-64)$\times 10^4$ g/mol and polydispersity index of 2.2.

The analysis reveals that samples containing additives initiate thermal decomposition earlier than pure AP- the incorporation of PFDMS reduces AP's decomposition temperature by approximately 100°C. Notably, the polymeric ferrocene catalyst demonstrates superior efficiency compared to free ferrocene. Furthermore, while the molecular weights of polymeric catalysts vary significantly, this difference shows minimal impact on their thermal decomposition behavior.

The role of iron content in polymeric catalysts

Thermal decomposition studies of propellants containing grafted HTPB-Fc revealed two distinct decomposition peaks at 500°C and 930°C, corresponding to: a) Mass loss through depolymerization, b) Decomposition of the grafted polymer.

Propellants incorporating HTPB-Fc with 0.45 wt. % iron demonstrate: a) 50% increase in burning rate versus baseline propellant, b) Preservation of mechanical properties, c) maintained processing characteristics (Fig. 12).

Comparative studies confirm iron in HTPB-Fc exhibits significantly higher catalytic efficiency than conventional catalysts like iron oxide and copper chromate.

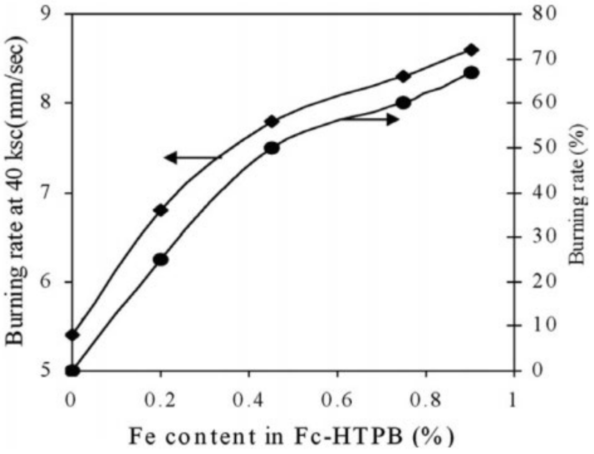


Fig. 12. Fuel combustion rate diagram of Fc-HTPB as a function of propellant combustion rate and iron content in the fuel.

The role of the branching structure of the catalyst in its migration

In a study conducted in 2010, Xiao used ferrocene modified highly branched polyamine as a combustion catalyst in HTPB based propellant for catalyst migration (Fig. 13). It can be seen that sample (c), which has a highly branched structure, has the lowest migration.

In this study the migration behavior of a ferrocene-modified hyperbranched polyamine, when used as a burning-rate catalyst in HTPB-based propellants, has been investigated. The research focuses on understanding the movement of the ferrocene-modified catalyst within the propellant matrix during combustion. This study aims to optimize the catalyst's effectiveness by analyzing its position and concentration relative to the burning surface.

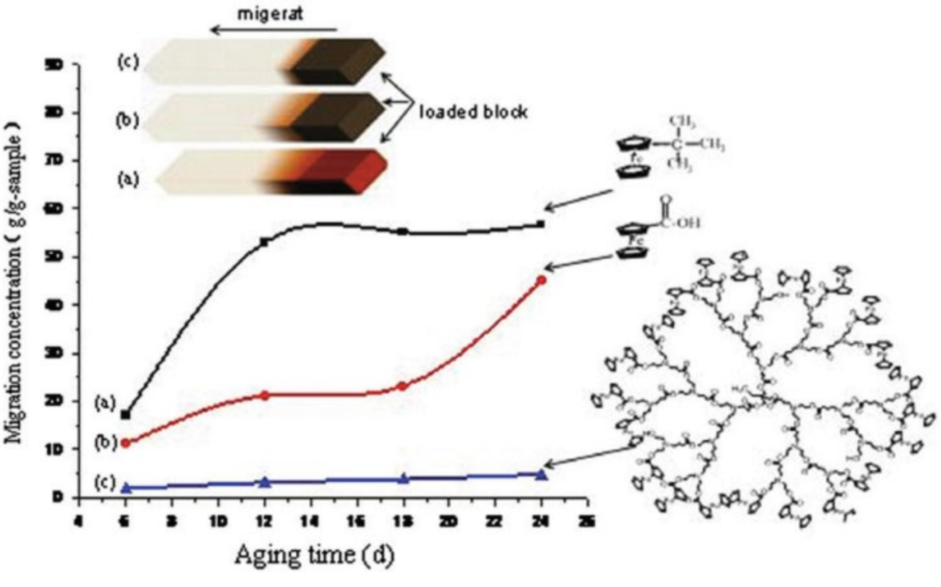


Fig. 13. Changes in concentration of a) tert-butylferrocene, b) ferrocenecarboxylic acid, and c) dendrimeric macromer based on ferrocene-modified polyamines (NBHE-Fc) during aging of hydroxyl-terminated polybutadiene at 60°C.

Polymeric inorganic and hybrid combustion catalysts

Ferrocene-containing polymeric catalysts based on butacene (BTC) were synthesized and evaluated. Initial studies focused on its application in HTPB blends, followed by combined use of butacene with transition metal oxide catalysts – iron oxide(Fe_2O_3) and copper chromite $(CuCr_2O_4)$ – in

non-aluminized HTPB-based propellants.

Key findings from table 2 (burning rates of butacene-containing propellants), Replacement of 25% HTPB with butacene resulted in 100% increase in burning rate within 2-3 MPa pressure range.

Table 2. Burning rate of propellants containing butacene.

HTPB compared to butacene	Combustion rate (mm/s) at pressure (MPa)					
	2	3	5	7	9	n
1 : 0	2.8	6.11	7.14	9.16	9.17	0.53
3 : 1	1.18	9.20	1.23	5.26	2.28	0.29
1 : 1	7.19	4.23	5.26	6.32	2.33	0.30
1 : 3	7.23	1.27	8.30	6.32	7.33	0.22
0 : 1	0.24	3.37	8.30	9.23	0.34	0.23
Basic composition: 22 % binder, 78 % AP (4 µm); binder composition: ratio of HTPB or butacene to dioctyladipinate (DOA) (45 : 55) with toluene diiscyanite (TDI) (NCO:OH = 1:1)						

An HTPB-based catalyst is presented, which causes a significant increase in the burning rate (Table 3). It can be seen that as its amount decreases, the efficiency of the inorganic catalyst also decreases. It is evident that the hybrid catalyst performs better than the polymer-based one alone, although the activity of the inorganic catalyst is much lower than that of the butadiene-based ferrocene polymer catalyst [30]

Table 3. The influence of ballistic modifiers on the Fc-HTPB propellant.

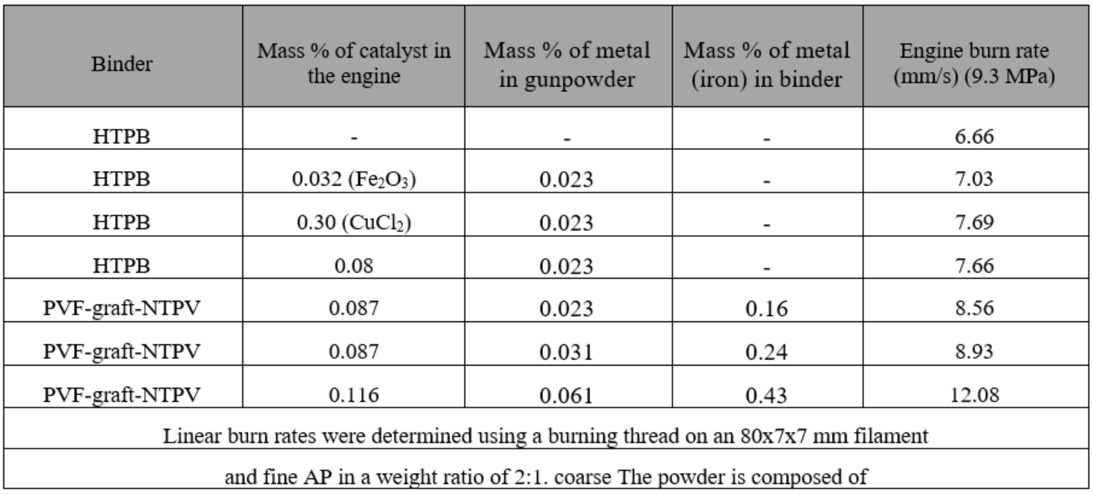
Ratio HTPB : BTC	BMs (p)	Combustion rate (mm/s) at pressure (MPa)					
		2	3	5	7	9	n
1 : 0	-	2.8	6.11	7.14	9.16	9.17	0.53
1 : 0	CC (3)	1.17	1.21	1.26	5.29	3.31	0.41
1 : 0	FO (3)	2.17	2.22	3.28	8.31	6.32	0.43
1 : 1	-	7.19	4.23	5.26	6.29	2.31	0.30
1 : 1	CC (3)	5.24	9.28	7.30	8.31	4.38	0.22
1 : 1	FO (3)	8.26	1.29	3.31	8.32	9.38	0.22
0 : 1	-	2.27	9.31	3.36	5.38	6.39	0.23
0 : 1	CC (3)	8.28	4.39	5.40	2.41	9.42	0.22
0 : 1	FO (3)	3.32	7.39	1.41	1.41	2.46	0.21
Basic composition: 22 % binder, 78 % AP (µm). Binder composition: HTPB or butacene to DOA ratio (45) to TDI (NCO:OH = 1:1							

Burning rate enhancement using grafted copolymer catalysts

The synthesized graft copolymer of HTPB and polyvinylferrocene (PVF) demonstrated significant burning rate enhancement in HTPB-based propellants. The catalytic efficiency showed direct correlation with the degree of PVF grafting - higher PVF content yielded greater combustion acceleration. Chemically-bound ferrocene moieties within the polymer backbone proved particularly effective, as the catalytic sites became integral to the macromolecular structure[31]

Aluminium-free propellants are formed from PVF-graft-HTPB with 84.6% AP as binder (coarse to fine fraction ratio 1:2), trimethylolpropane and toluene diisocyanates as condensation binders in stoichiometric ratio of NCO: OH = 1:1 and dibutylol dodilaurate as catalyst. As can be seen from the data in Table 4, the attached PVP shows better performance compared to other metallic and non-polymeric catalysts. In addition, the increase in combustion rate increases when the amount of PVF attached to the HTPB chain increases [31, 32].

Table 4. Motor test burning rates for various catalyst types and loadings.



Binder	Mass % of catalyst in the engine	Mass % of metal in gunpowder	Mass % of metal (iron) in binder	Engine burn rate (mm/s) (9.3 MPa)
HTPB	-	-	-	6.66
HTPB	0.032 (Fe_2O_3)	0.023	-	7.03
HTPB	0.30 ($CuCl_2$)	0.023	-	7.69
HTPB	0.08	0.023	-	7.66
PVF-graft-NTPV	0.087	0.023	0.16	8.56
PVF-graft-NTPV	0.087	0.031	0.24	8.93
PVF-graft-NTPV	0.116	0.061	0.43	12.08
Linear burn rates were determined using a burning thread on an 80x7x7 mm filament				
and fine AP in a weight ratio of 2:1. coarse The powder is composed of				

Migration resistance and anti-migration strategies

Combustion catalysts in solid propellant composites exhibit migration during storage, leading to uneven burning distribution and compromised motor performance. This phenomenon particularly affects ferrocene-containing polymers, which undergo direct migration due to high concentration gradients within the system (Fig. 14) [33].

Two methods can be used to improve the migration characteristics of ferrocene-based combustion catalysts.

A - Introduction of hyperbranched copolymers incorporating PCs, which increases their molecular weight and reduces their migration properties

B- Synthesis of ferrocene derivatives having a crosslinked structure, which can be achieved by introducing active ferrocene-containing functional groups during crosslinking (Figure 16) [34, 35].

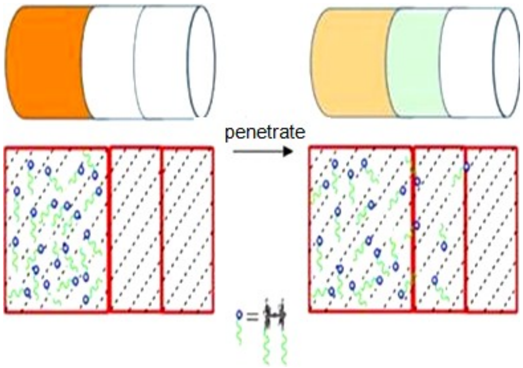


Fig. 14. Penetration of FA derivatives into fuels.

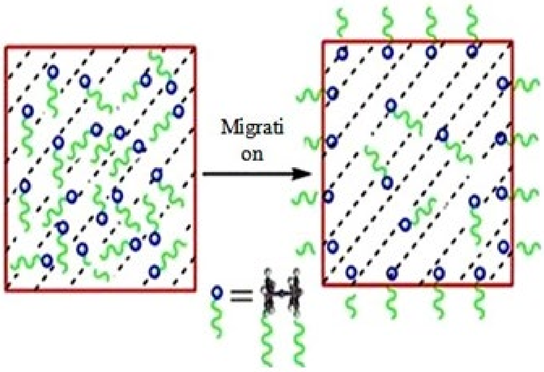


Fig. 15. Possible mechanism of migration of PC-derivatives from outside to the surface powder.

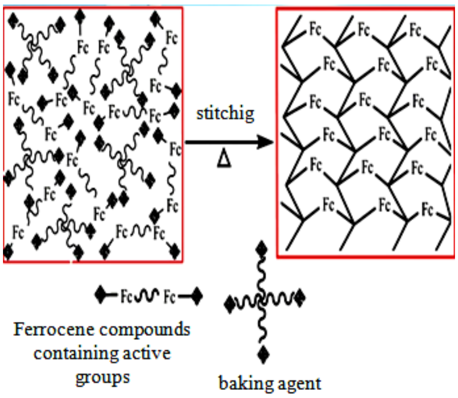


Fig. 16. Formation of a mesh containing ferrocene derivatives with by functional group.

4. CONCLUSIONS

This article has reviewed the types and migration properties of ferrocene-based polymer burning catalysts ffective catalysts widely used in solid rocket propellants. Although significant progress has been made in the development of burning catalysts for solid fuels, catalyst migration within the propellant remains a critical issue. This migration reduces the burning rate, affects the predictability of the combustion process, and also deteriorates the physicomechanical properties of the solid propellant. The tendency to migrate is influenced by factors such as the structure and amount of the additive, as well as its solubility (compatibility) and molecular mobility.

To mitigate these challenges future research should focus on developing novel ferrocene derivatives with enhanced structural stability and reduced mobility. Incorporating polymeric matrices or covalent bonding strategies could help anchor the catalyst molecules more effectively, minimizing undesired migration. Additionally, advanced characterization techniques, such as molecular dynamics simulations and high-resolution microscopy, could provide deeper insights into migration mechanisms at the nanoscale. Further experimental studies should also explore the effects of environmental conditions, such as temperature and humidity, on catalyst stability over long-term storage. By addressing these factors, researchers can design more efficient and reliable burning catalysts, ultimately improving the performance and safety of solid rocket propellants.

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