

Testing of LiAlH_4 as a Potential Additive to Paraffin Wax Hybrid Rocket Fuel

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This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.



Vince Caponpon
SMC/EAF

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14. ABSTRACT The burn rate of paraffin wax fuel was measured in air with and without addition of LiAlH ₄ . The paraffin wax was mixed with 7–24% LiAlH ₄ (lithium aluminum hydrate) by weight. The wax was then molded into candles. The candles were lit in air, and the mass loss with time was recorded. The 24% LiAlH ₄ was found to enhance the burn rate of the candles by a factor of ~14. LiAlH ₄ was found to be a suitable compound to consider as a potential additive to hybrid rocket motors. The paraffin wax/LiAlH ₄ was stable under conditions where the wax was solidified, re-melted, and molded at 70°C. The hydrophobic paraffin wax was found to isolate the LiAlH ₄ from reaction with both water vapor and upon submerging in liquid water. The paraffin wax/LiAlH ₄ candles stored for over a month under atmospheric conditions were lit and burned similarly to freshly made candles. The addition of LiAlH ₄ to paraffin wax could significantly increase the burn rate of the paraffin wax used in a hybrid motor.					
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1. Introduction

Hybrid rocket motors have the potential to replace current solid rocket motors' functionality as boost phase launch vehicle components. Hybrid rockets have several advantages to current solid rocket motors based on ammonium perchlorate. Hybrid rocket motors are non-toxic, non-hazardous, shippable as freight cargo, and potentially carbon neutral;¹ and they can be throttled for thrust control or shut down in case of an on-pad anomaly and restarted on demand. Since the fuel is non-explosive, the fuel can be fabricated on-site and thus can save cost in both manufacturing and launch operation.^{2,3} The main downside to conventional hybrid motors based on high-density, long-chained polymers such as HTPB (hydroxyl-terminated polybutadiene) is that they have very low regression rates.^{4,5} To use these materials as hybrid rocket motors, the fuel must utilize a multi-port grain to enhance burn rate. However, these multi-port designs degrade the overall performance and cost effectiveness of the hybrid propulsion system.

Paraffin wax-based hydrocarbon fuels have shown regression rates 3 to 4 times higher than conventional polymeric hybrid fuels.^{2,6} Use of the high-regression-rate, paraffin-wax-based fuel eliminates the need for multiple port fuel grains without resorting to regression rate enhancing additives. The paraffin fuels have demonstrated specific impulse performance and favorable density impulse performance comparable to hydrocarbon- fueled liquid systems.^{7,8} Several small-scale paraffin wax hybrid rockets have been tested in recent years.⁷⁻¹² These paraffin hybrid rockets to date have utilized fuel grain sizes up to 11 inches in diameter. To reach commercial launch vehicle sizes for use in payload delivery, larger rockets will be needed. Upon scaling up current paraffin wax hybrid motors, the use of additives may be needed to achieve more efficient burn rates in large-diameter fuel grains.

While burn rates for paraffin wax hybrids are large compared to other hybrid fuels, additives can be used with paraffin wax to further tailor the regression rate and specific impulse (Isp). Traditional solid rocket motor additives such as aluminum have previously been tested.^{8,13} Adding aluminum to paraffin was found to increase the combustion temperature and regression rate. Aluminizing the paraffin can also decrease the optimal oxidizer-to-fuel ratio, making it possible to reduce the volume of the oxidizer tank relative to straight paraffin hybrid rockets.⁸ Another potential additive, lithium aluminum hydride (LiAlH_4), has not yet been tested as an additive to paraffin wax. Metal hydrides have previously been tested as an additive for other non-paraffin wax-based hybrid rocket fuels.¹⁴ The major difficulty (and advantage) of metal hydrides is that they are very reactive with water and therefore humid air. Metal hydride compounds need to be stored in a dry environment. In addition, most conventional binders, such as hydroxyl-terminated polybutadiene (HTPB), will either react with the metal hydride or allow water to get at the metal hydride powder in the binder.¹⁴ The addition of LiAlH_4 has previously been shown to increase the theoretical Isp and regression rate of the hybrid rocket fuel cyclopentadiene, although the tests found low combustion efficiency.¹⁵

This study will look at the potential for LiAlH_4 as an additive for paraffin wax-based hybrid rocket fuels. The LiAlH_4 will be added to paraffin wax and tested for compatibility with water. Paraffin

wax/ LiAlH_4 was found to be stable while exposed to air. Paraffin wax/ LiAlH_4 was found to be storable under atmospheric conditions and was even stable when submerged in water. The paraffin wax/ LiAlH_4 was found to be both re-meltable and moldable. The paraffin wax/ LiAlH_4 was burned in air with a wick and compared to straight paraffin wax burning. Overall, the mass loss rate of paraffin wax with LiAlH_4 added was found to be greater than pure paraffin wax when burned with an open flame.

2. Experimental

A low-purity paraffin wax with a melting point between 58°C and 62°C was selected for use in the candle manufacturing. This wax had chemical properties similar to the paraffin wax used in the most advanced paraffin wax hybrid rocket motors.¹⁻³ The lithium aluminum hydride (LiAlH_4) powder purity was >95 %. The LiAlH_4 decomposes at 125°C, which is considerably higher than the melting temperature of the paraffin wax. The LiAlH_4 was added to the paraffin inside a nitrogen purge bag. A sample of the wax (which comes from the manufacturer in small chunks) was weighed and then placed into a Pyrex beaker. The Pyrex beaker was placed on top of a hot plate inside the nitrogen purge bag. The hot plate was set to 70°C to ensure 100% melting of the wax, but not cause decomposition of the LiAlH_4 . The LiAlH_4 was weighed inside the nitrogen purge bag. The LiAlH_4 was then added to the molten wax, which was then stirred in order to get a uniform distribution of the LiAlH_4 . The hot plate was then turned off, and the wax was allowed to cool. The solidified wax was then removed from the nitrogen purge bag.

Four separate paraffin wax- LiAlH_4 mixtures were made (0%, 7%, 20%, and 24% by weight). The paraffin waxes were melted on a hot plate set at 70°C. The molten wax was then poured into a candle mold (Figure 1). The candle mold has four bolts that hold the two halves together. The molds are 9.8 mm in diameter and 50.6 mm long. The wick stand (as seen in Figure 1) fits into the mold and holds the wick in place during pouring. The candles are removed by unscrewing the mold.

The candles are placed on an electronic balance inside a fume hood. A copper disk (33.20 g) was placed under the candle to prevent damage to the scale from heating by molten wax. The candle wick was then lit by a match. The weight was recording by a video camera that captured both the burning

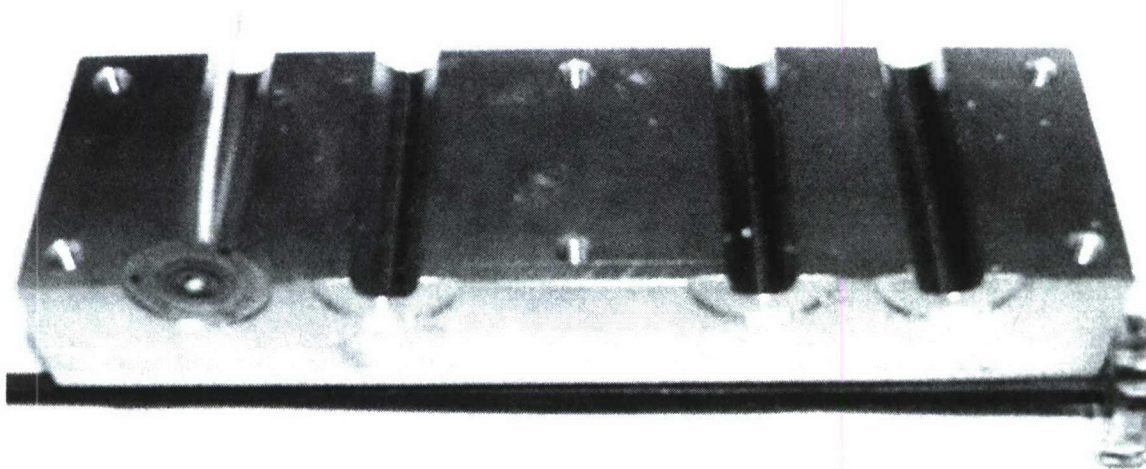
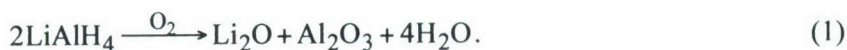


Figure 1. Half of the candle mold used to manufacture paraffin wax candles. The wick and wick stand are shown in place in the left-most candle mold.

of the candle and the weight displayed on the balance. In most cases, the experiment ended when the candle completely melted and the wick went out. This took from 2 to 8 min depending on the candle used.

The burning of the candle wax produces primarily gaseous products: carbon dioxide (CO₂) and water vapor (H₂O). The burning of the LiAlH₄ produces aluminum oxide (Al₂O₃) and lithium oxide (Li₂O). These two solid products increase the mass of the candle upon burning.



The metal oxide products are 1.73 times more massive than the LiAlH₄ that has been burned away. Thus, the mass observed at the end of the burning experiment includes the solid oxidized waste. This waste product can be determined to get the actual total mass burned. The observed mass loss was used to determine the total mass loss from Eq. (2). The mass lost from combustion is the mass loss observed on the balance + the mass gained from metal oxide. The mass loss observed (mass_{obs}) from the candle includes the oxidized paraffin and the oxidized hydrogen from the LiAlH₄ (10.5% of the LiAlH₄ mass). The oxides of lithium and aluminum increase the mass by 1.73 from LiAlH₄. Thus, the total mass loss is related to the mass loss observed by Eq. (2).

$$\text{mass}_{\text{total}} = \text{mass}_{\text{obs}} + (\text{mass}_{\text{obs}} - \text{mass}_{\text{obs}} \times \%_{\text{LiAlH}_4} \times 0.105) \times \%_{\text{LiAlH}_4} \times 1.73. \quad (2)$$

The stability of the paraffin wax/ LiAlH₄ mixtures upon exposure to water was tested. The wax was placed into a beaker filled with 0.5 ml of water. A thermal couple was used to record the temperature increase of the water as the LiAlH₄ reacted with the water. The increase in the temperature of the water can be predicted. In water, the LiAlH₄ should react as follows:



The enthalpy of the reaction (ΔH_r) is −982 kJ mol^{−1}. The change in the internal energy (ΔU) of the system is equal to

$$\Delta U = \Delta H - nRT. \quad (4)$$

The rise in the temperature of the beaker should then be equal to

$$\Delta U = C_v dT, \quad (5)$$

where C_v is the heat capacity of the water (4 J K^{−1} g^{−1}).

3. Results

Figure 2 shows two candles burning. The right candle is a paraffin wax candle, while the left candle is a mixture of paraffin wax and LiAlH_4 (20%). Figure 2 clearly shows the additive was observed to increase the burn rate of the paraffin candle. However, it also shows that the paraffin wax melts faster with the additive. It's not clear whether the candle is actually losing mass faster or simply melting faster. Figure 3 shows the mass loss with time for a burning candle with and without 24% LiAlH_4 added. The first minute of burning shows similar mass loss for the two separate candles. However, after 1.5 min, the candle with the additive has considerably accelerated mass loss compared to the pure paraffin wax candle. The initiation time was observed for all the candles with the additive. The initiation time includes time for the wick to burn down to the bulk candle. Most likely this initiation time reflects the time needed for the candle to heat up beyond the temperature where LiAlH_4 actively begins to decompose (125°C). Figure 3 clearly demonstrates that the additive increases the mass loss rate of the burning candle.

The candles all have slightly different initial candle weight; thus, the mass loss rate needs to be scaled by the initial mass. The average initial candle weight was 4.15 g. Figure 4 has the percent of initial mass loss observed over the burning time period. The pure paraffin wax candle burn rate was observed to be similar to the 7% LiAlH_4 . However, there was a considerable increase in the burn rate with 20–24% LiAlH_4 added. Table 1 has the peak scaled mass loss rate observed for each trial of the paraffin wax/ LiAlH_4 mixture. The peak mass loss rate from the 20–24% LiAlH_4 candles was ~ 14 times greater than the 0–7% LiAlH_4 candles.

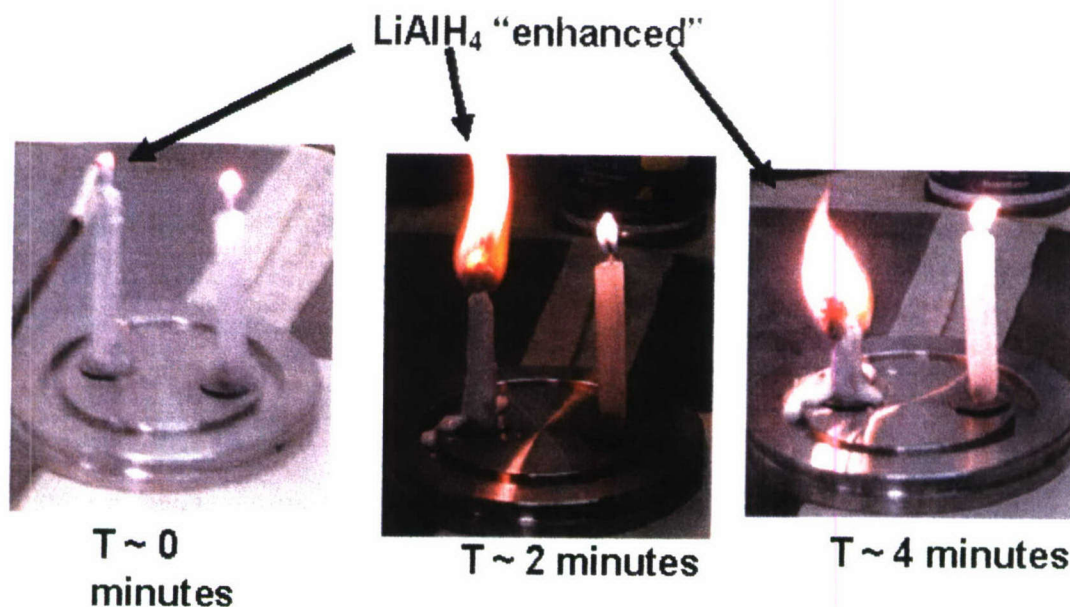


Figure 2. Two paraffin wax candles burning. The left candle has been doped with 20% LiAlH_4 .

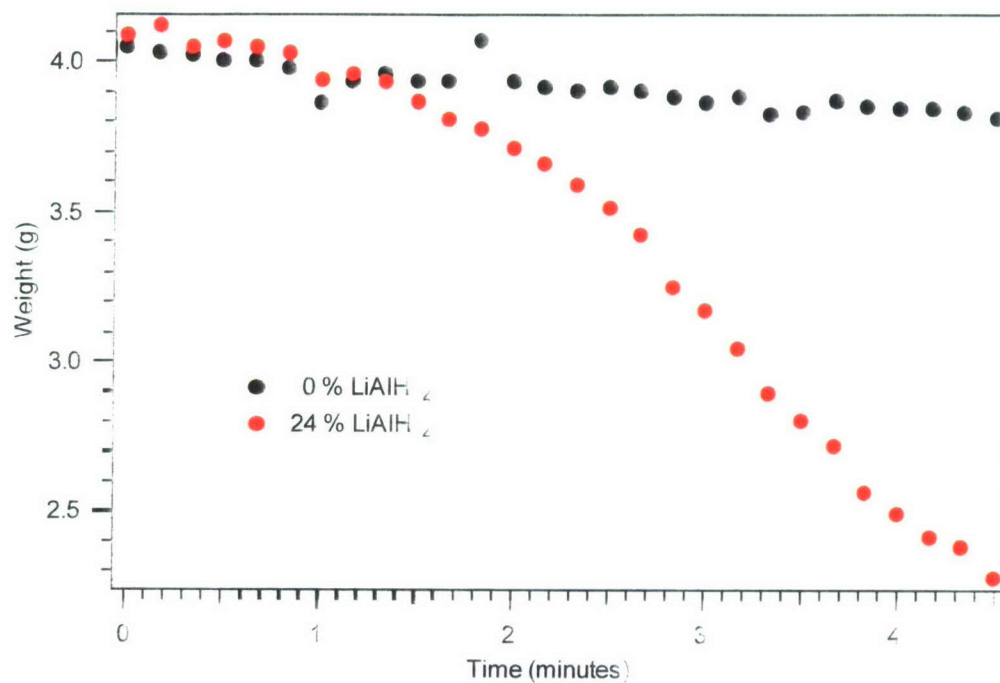


Figure 3. Observed mass loss as a function of time for the paraffin wax candles. The black (●) candle has 0% LiAlH₄ while the red (●) has 24% by weight LiAlH₄.

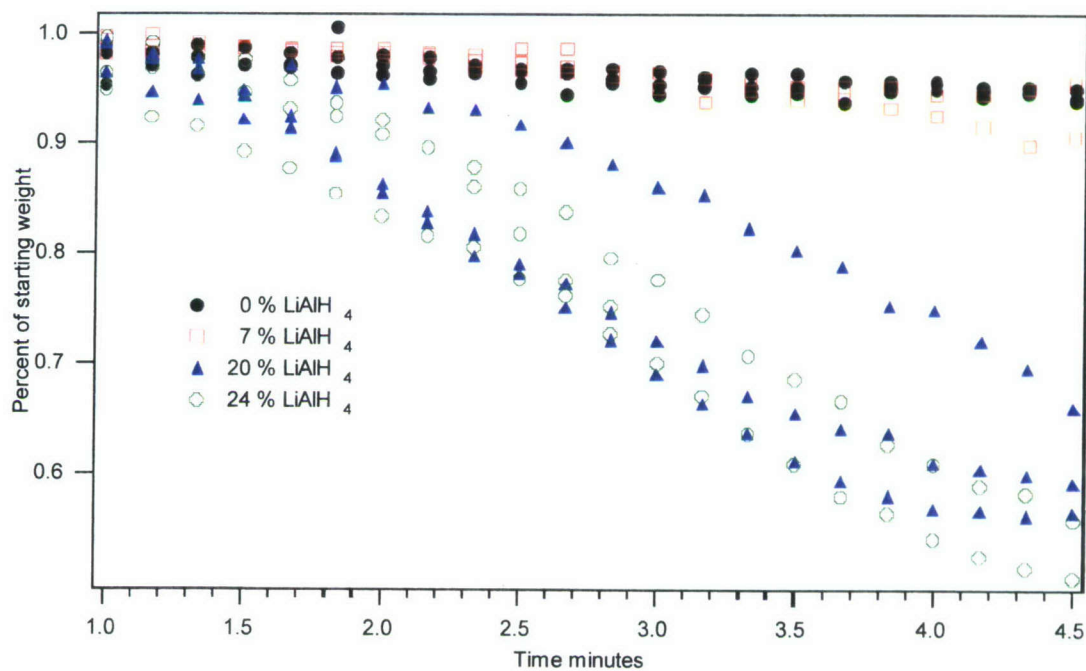


Figure 4. Observed mass percent loss as a function of time for the paraffin wax/LiAlH₄ candles. The black (●) candle has 0% LiAlH₄, the red (□) has 7% by weight LiAlH₄, the blue (Δ) has 20% by weight LiAlH₄, and the green (○) has 24% by weight LiAlH₄.

Table 1. Peak Mass Loss Rate (g min^{-1}) of the Paraffin Wax/ LiAlH_4 Candles. The mass loss has been corrected for the lithium and aluminum oxide accumulated on the balance during the test *via* Eq. (2).

Composition (% LiAlH_4)	Observed mass loss (g min^{-1})	Determined by Eq. (2) (g min^{-1})	Average of 3 trials (g min^{-1})
0	0.047	0.047	0.048
0	0.049	0.049	
0	0.047	0.047	
7	0.069	0.077	0.075
7	0.045	0.050	
7	0.086	0.097	
20	0.621	0.831	0.736
20	0.541	0.724	
20	0.487	0.652	
24	0.874	1.228	0.959
24	0.540	0.758	
24	0.630	0.890	

The second and third run were conducted ~5 weeks apart. The candles for the third run were stored for 5 weeks exposed to the atmosphere. The mass loss rates for the 2nd and 3rd run were similar for both the 20% and 24% LiAlH_4 candles. The LiAlH_4 does not appear to have been significantly reacting with atmospheric water vapor. This indicates that the hydrophobic paraffin wax provides adequate insulation of the LiAlH_4 to allow long-term storage.

The paraffin wax/ LiAlH_4 exposed directly to water showed very little reactivity. Table 2 shows the increase in temperature of 0.5 ml water upon exposure to 0.1–0.15 g of LiAlH_4 . Some of the LiAlH_4 samples were mixed in the paraffin wax, while others had no wax matrix. The samples without paraffin wax showed much greater temperature rise compared to those inside the paraffin wax. About 0.8% of the predicted heat rise was observed for the paraffin wax/ LiAlH_4 samples. For the pure LiAlH_4 , the water temperature raised to only about 55% of the predicted value (the rest of the heat presumably was lost to either the atmosphere or the glass container). Including the heat loss to the container, about 1.5% of the energy content of the paraffin wax/ LiAlH_4 was released when the wax was submersed in water. Given that paraffin wax is very hydrophobic, the most likely reaction site for the samples was near the exposed surface. The surface-to-volume ratio of the samples was ~40%.

Table 2. Observed Increase in Temperature of 0.5 ml of Water Upon Exposure to Paraffin Wax/ LiAlH_4 Mixtures

Sample composition (% LiAlH_4)	Mass of LiAlH_4 in sample used	Measured temperature rise (K)	Predicted temperature rise (K)	Measure temperature/ Predicted temperature
100	0.10	40.2	67.3	0.60
100	0.09	30.6	60.6	0.50
20	0.14	0.2	94.3	0.002
20	0.15	0.8	101.1	0.008
20	0.12	1.2	80.3	0.015

The heat evolved was much less than 40%. The expected heat evolution from the samples observed would indicate that only a very small layer of paraffin is enough to isolate the LiAlH_4 from water. The results indicate that paraffin wax/ LiAlH_4 mixtures should be stable for long times at atmospheric conditions and should not self ignite upon exposure to water. For additional protection for long-term storage or shipping, a small layer of pure paraffin wax should adequately protect the doped wax from exposure to liquid water.

5. Conclusion

The potential for the use of LiAlH_4 as an additive for paraffin rockets has been examined in this study. The paraffin wax/ LiAlH_4 was found to be stable while exposed to air. The paraffin wax/ LiAlH_4 was found to be both re-meltable and moldable. Overall, the peak mass loss rate of paraffin wax with LiAlH_4 added was found to be ~ 14 times greater than pure paraffin wax. LiAlH_4 was found to have a high potential to be a beneficial additive to paraffin wax-based hybrid systems.

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