



Compatibility Testing – What is it really telling us?

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STANAG 4147

- Background
- Historical inspirations
- Limitations



EXPERIMENTAL RESULTS

- Materials and compatibility tests
- Compatibility with M7
- Variation of grain size

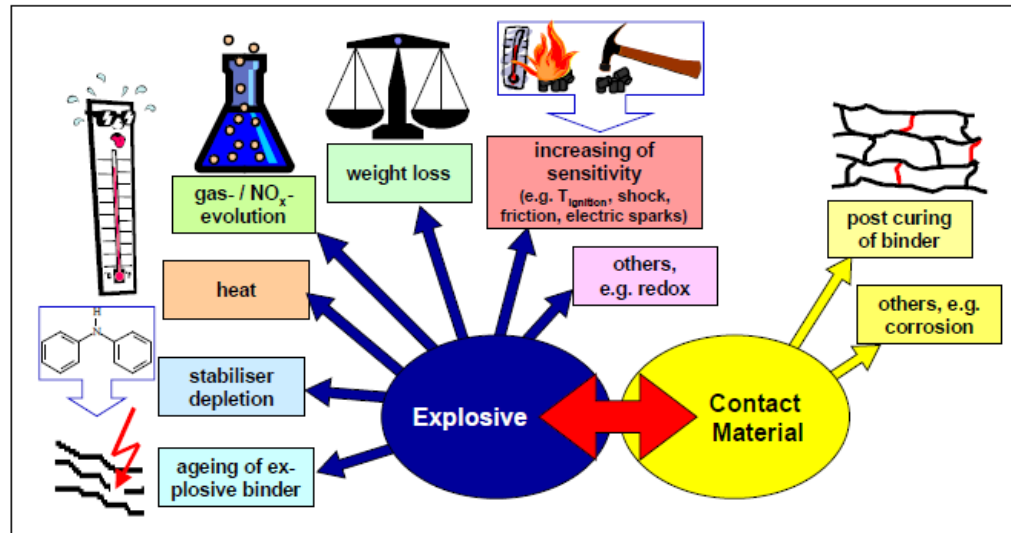


CONCLUSIONS



Background – the Why?

- Compatibility testing : before the Word War II
- Incompatibilities : chemical or physical reactions between EM and contact materials which could alter the service life of a munition
- Chemical compatibility: property of not having a significantly degraded chemical stability by being in contact with another energetic or inert material



VOGELSANGER and SOPRANETTI, Chemical Compatibility of Propellants with Contact Materials - Overview Possibilities, Limitations and Pitfalls, 2003

What is compatibility testing ?

- First definition : Marjorie St Cyr, Picatinny Arsenal (USA) in 1959

“if neither the explosive nor the polymer is adversely affected by direct contact, then the explosive-polymer combination is considered compatible”

Marjorie Saint Cyr, Compatibility of explosives with polymers, 1959

- AOP-38: 3 definitions for compatibility

“Absence of reactions between explosives and other components within a munition, leading to unacceptable changes in physical properties, sensitiveness or sensitivity of explosives in the munition.”

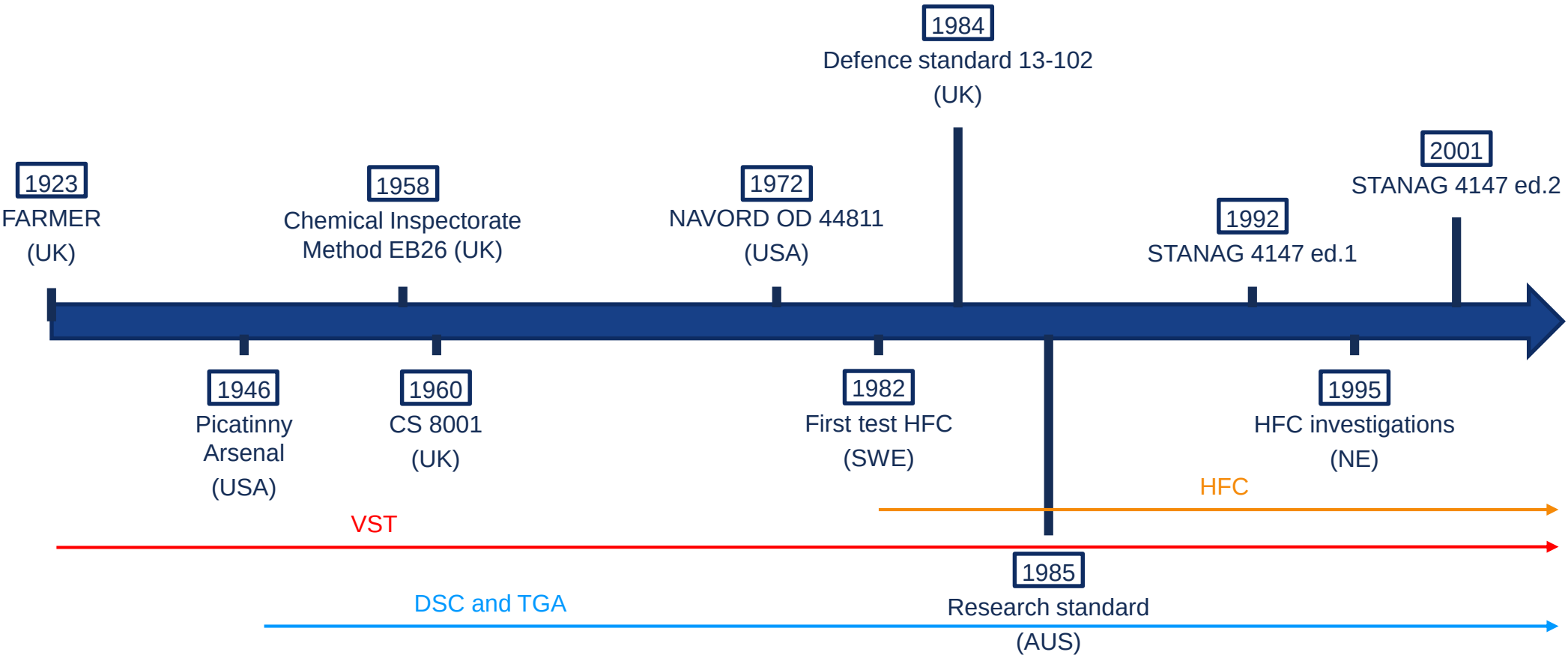
AOP-38

- STANAG 4147 : standardizes the procedure for compatibility testing and gives pass/fail criteria

“The purpose of a compatibility test in the context of this STANAG is to provide evidence that a material may be used in an item of ammunition without detriment to the safety or reliability of an explosive with which it is in contact or proximity”

STANAG 4147 ed.2

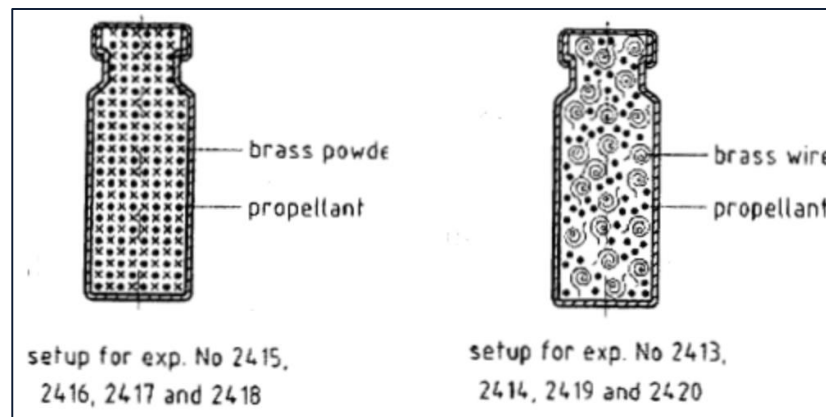
STANAG 4147 : historical context



Effect of grain sizes

- STANAG 4147 : between 0.2 mm and 2 mm except for DSC and TGA (smallest grain size required)
- Philosophy : increase the surface area between EM and the contact material
→ worst case of contact
- Contact not intended to reflect the reality

“For the NS34-10 propellant, the material was cut into sticks to fit the length of the HFC vial.”



TUNNEL, Evaluation of Techniques for Energetic Material
Compatibility Testing, 2020

WILKER and al, FROM BINARY MIXTURES TO COMPLETE AMMUNITION
COMPATIBILITY - A WHOLE CARTRIDGE IN ONE TAM AMPOULE – 2005

Experimental results : materials

- M7 (double based propellant)

Component	w/w (%)	Formula
NC, unspecified nitrogen content	54.600	
NGL	35.500	$C_3H_5N_3O_9$
Potassium perchlorate	8.050	$KClO_4$
Carbon black	1.200	
Ethyl Centralite	0.800	$C_{17}H_{20}N_2O$

- Contact materials:
 - PVC
 - Silicone
 - EPDM
 - Epoxy (cured)
 - Glue (DAREX OP 649F)



- DSC :**

- 2°C/min from 50°C to 500°C
- Grain size : < 0.2 mm
- 1 mg, mixing ratio 1:1

$$\Delta T = T_{\text{MaxC}} - T_{\text{MaxM}}$$

< 4°C
4°C ≤ ΔT ≤ 20°C or change in
peak shape
> 20°C

- TGA :**

- 2°C/min from 25°C to 550°C
- Grain size : < 0.2 mm
- 1 mg, mixing ratio 1:1

$$W_R = W_M - W_{M,calc} = W_M - (m_E * W_E + m_C * W_C) / (m_E + m_C)$$

< 4 %
4 % ≤ ΔT ≤ 20 %
> 20 %

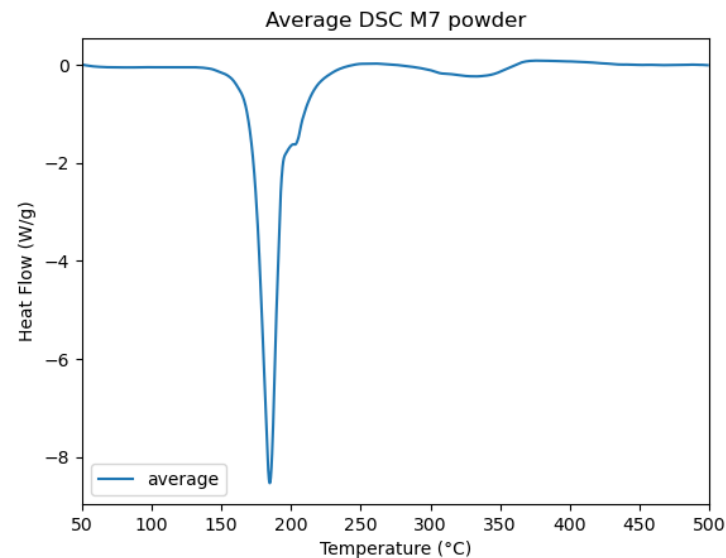
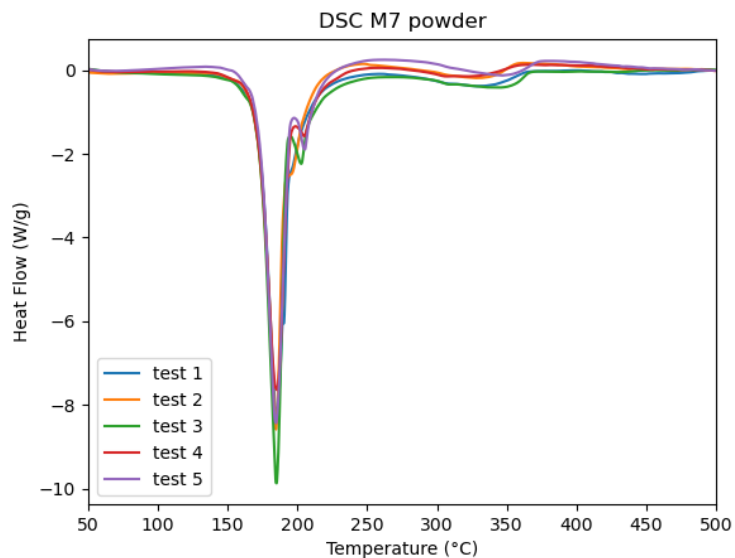
- HFC :**

- At 90°C for 3.43 days
- Grain size : between 0.2 mm and 0.63 mm
- Mixing ratio 1:1

$$Q_R = Q_M - Q_{M,cal} = Q_M - (m_e * Q_E + m_c * Q_C) / (m_E + m_C)$$

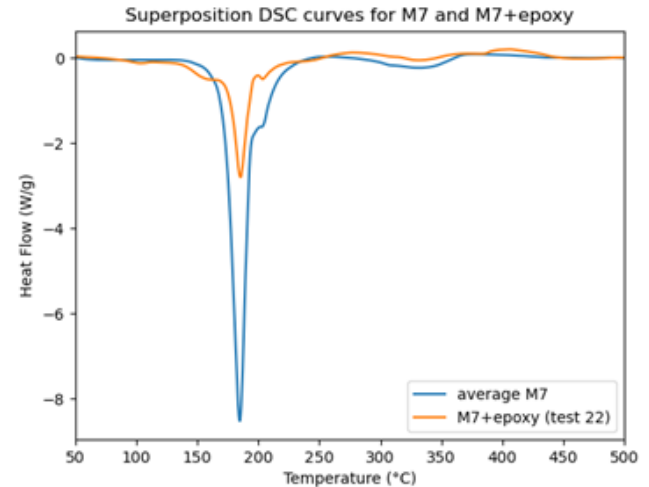
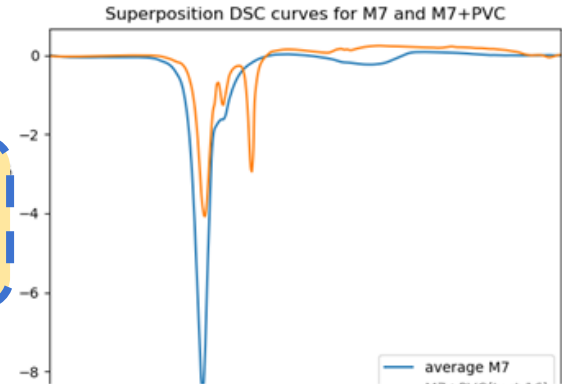
≤ 30 J/g
> 30 J/g

DSC results

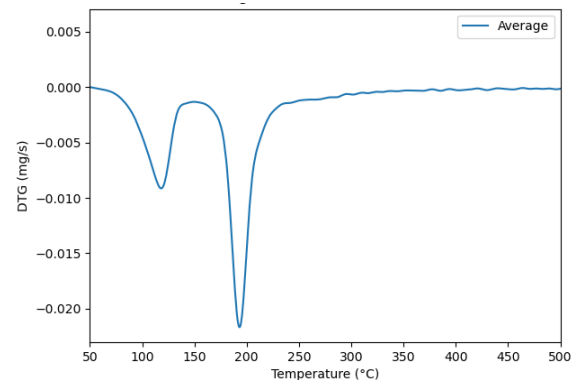
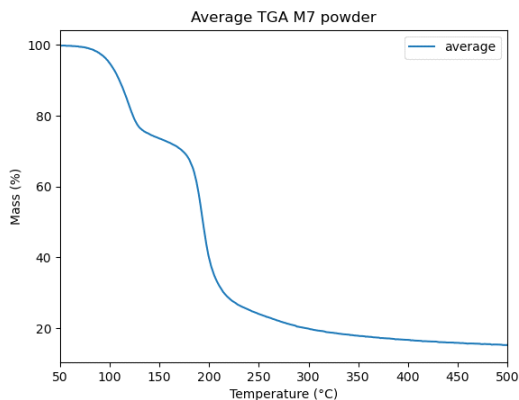
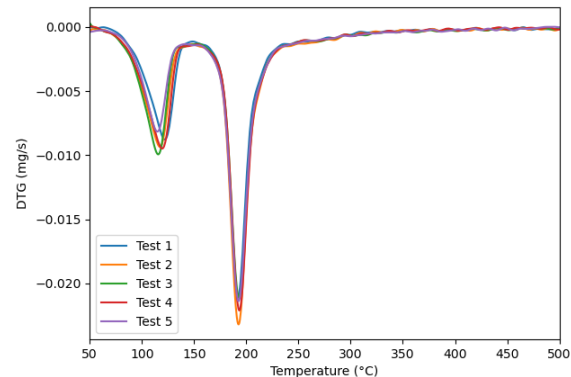
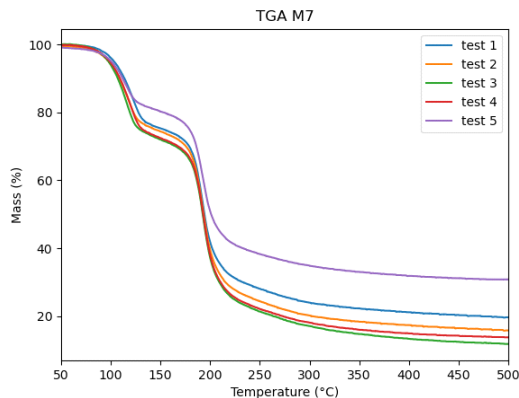


DSC results

Test N°	Test	Temperature of the first exothermal peak (°C)	ΔT (°C)	Compatibility
Average on all tests	M7	184,68	N/A	N/A
Test 16	M7 + PVC	186,72	-2,04	Change in peak shape
Test 17		187,43	-2,75	
Average		N/A	-2,395	
Test 18	M7 + silicone	184,97	-0,29	Compatible
Test 19		185,2	-0,52	
Average		N/A	-0,405	
Test 20	M7 + EPDM	187,05	-2,37	Compatible
Test 21		187,03	-2,35	
Average		N/A	-2,36	
Test 22	M7 + epoxy	185,48	-0,8	Change in peak shape
Test 23		187,15	-2,47	
Average		N/A	-1,635	
Test 24	M7 + glue	182,32	-2,36	Compatible
Test 25		182,84	1,84	
Average		N/A	2,1	

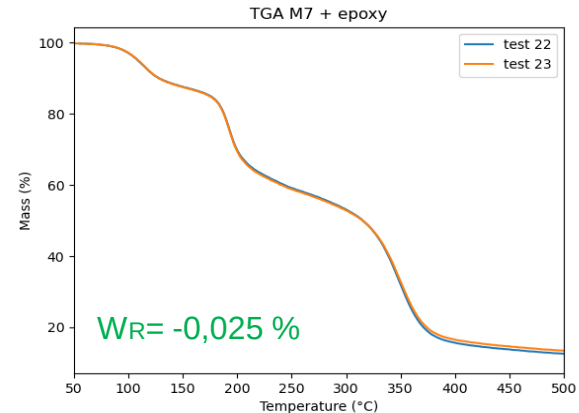
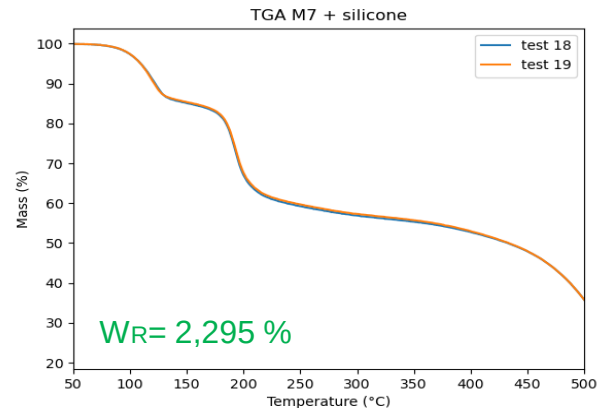
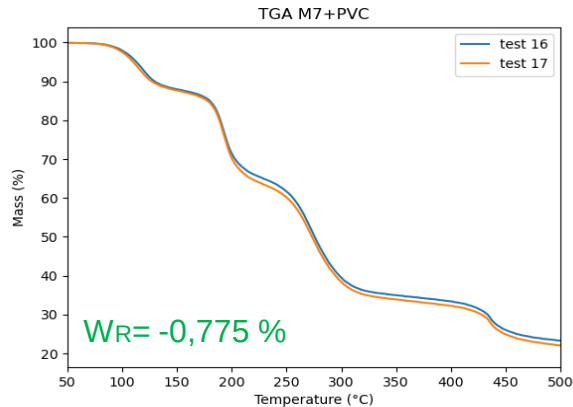
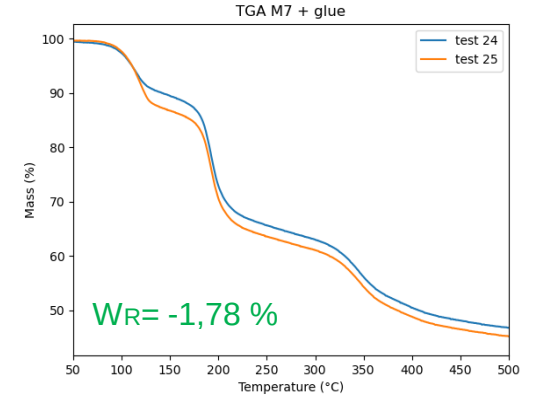
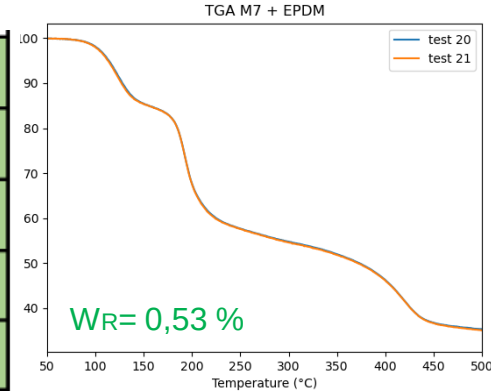


TGA results

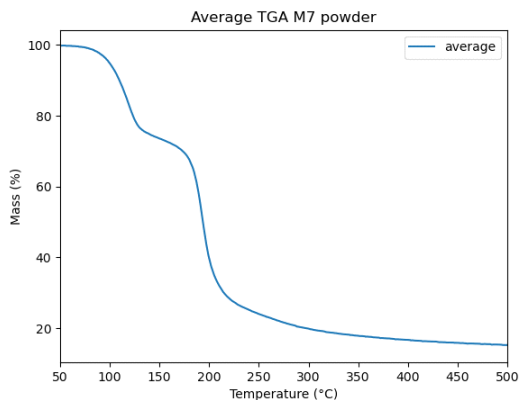
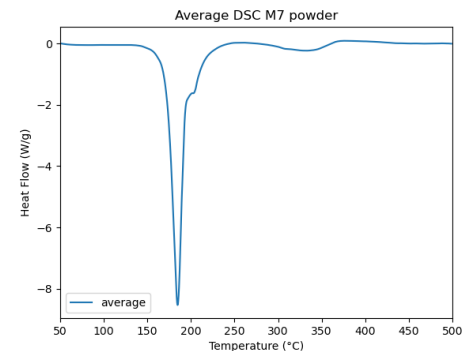
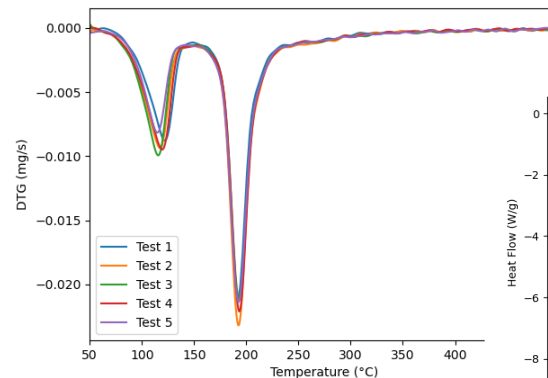
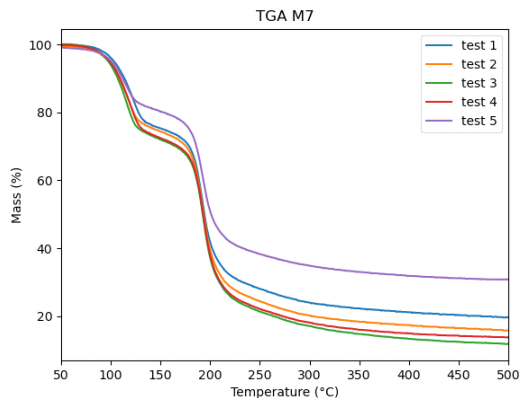


TGA results

Test 16	M7 + PVC	N/A	N/A	23,28	-0,49	Compatible
Test 17				24,16	-1,06	
Test 18	M7 + silicone	N/A	N/A	27,75	2,86	Compatible
Test 19				26,61	1,73	
Test 20	M7 + EPDM	N/A	N/A	26,73	0,54	Compatible
Test 21				26,82	0,52	
Test 22	M7 + epoxy	N/A	N/A	24,73	0,13	Compatible
Test 23				24,94	-0,18	
Test 24	M7 + glue	N/A	N/A	22,09	-3,01	Compatible
Test 25				24,52	-0,55	



TGA results



Component	w/w (%)	Formula
NC, unspecified nitrogen content	54.600	
NGL	35.500	$C_3H_5N_3O_9$
Potassium perchlorate	8.050	$KClO_4$
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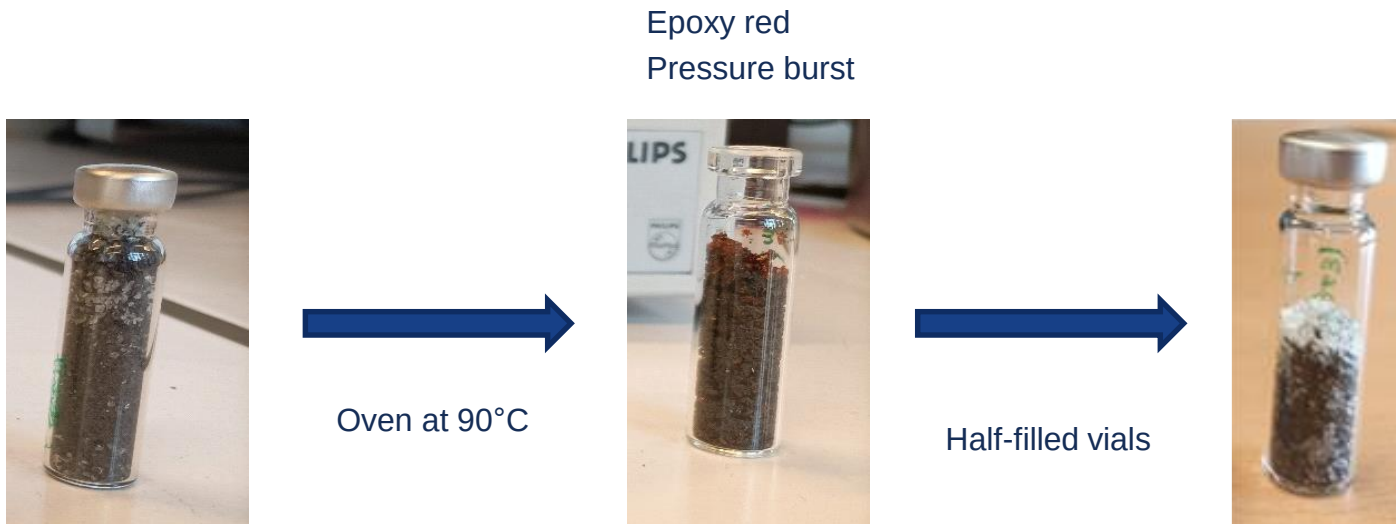
Experimental results : compatibility via HFC

- To avoid damaging the HFC, Sample vials were made up and placed in an oven at 90°C for 3,43 days → any significant reaction
- Replicates the HFC measurement conditions.

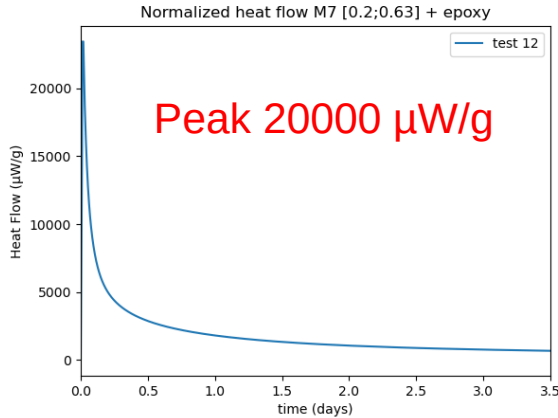
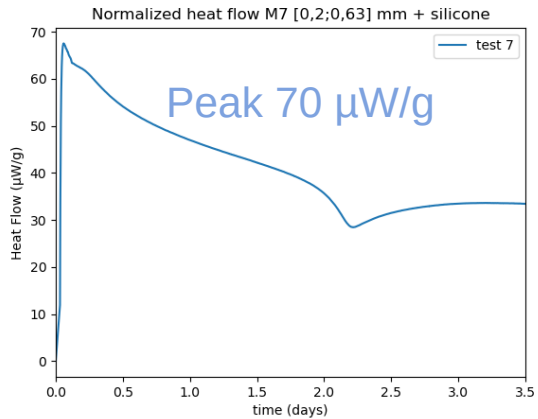
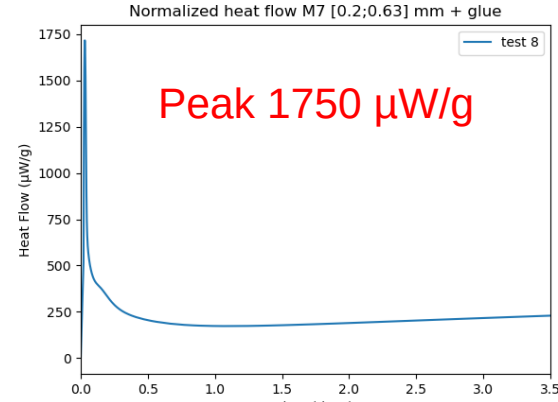
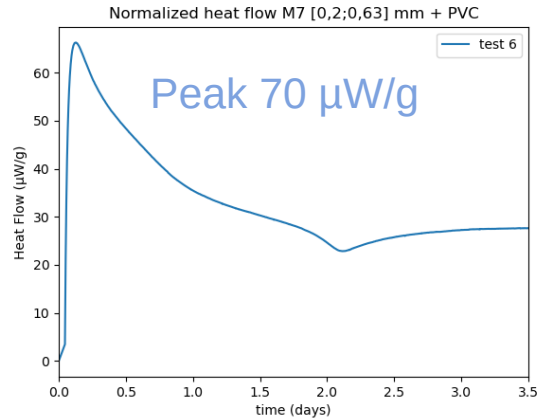


HFC results

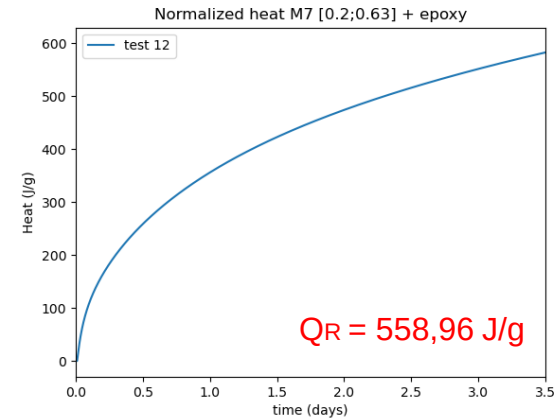
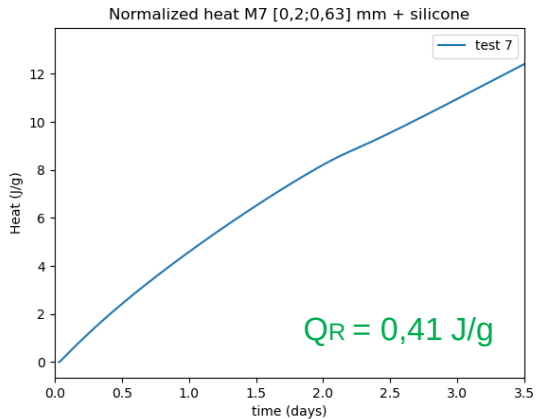
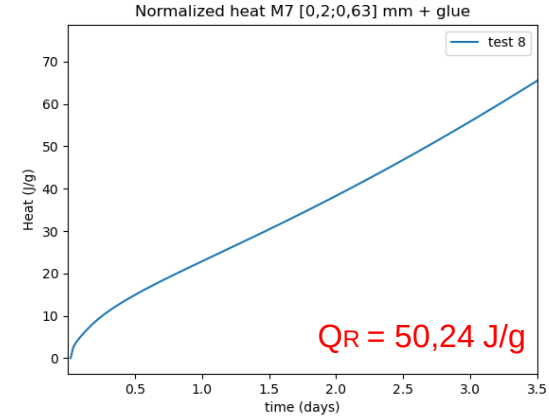
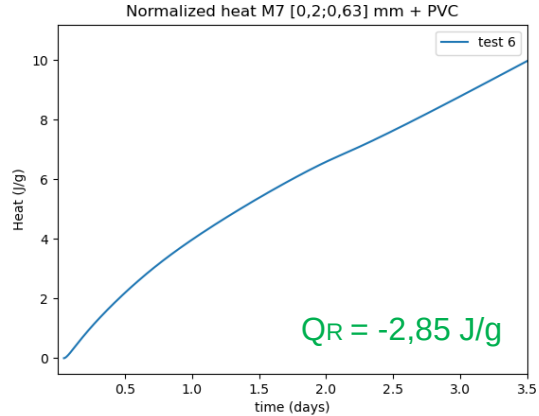
- Previous test: vials in an oven at 90°C for 3,43 days → any significant reaction



HFC results



HFC results



Experimental results : variation of grain size

- M7 initial shape (a)
- M7 diced up in squares [3;5] mm (b)
- M7 grinded [1;2] mm (c)
- M7 grinded [0,2;0,63] mm (d)

Contact materials:

- Silicone → compatible
- Glue → limit of compatibility
- Epoxy → incompatible



(a) Initial shape lenght cut



(b) Squares

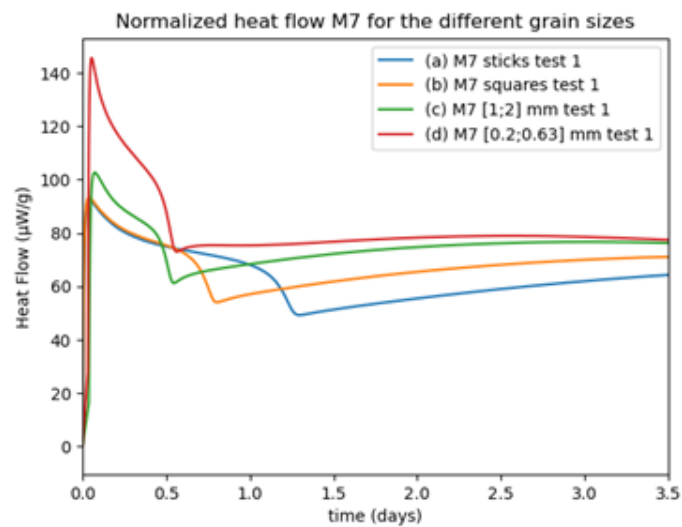
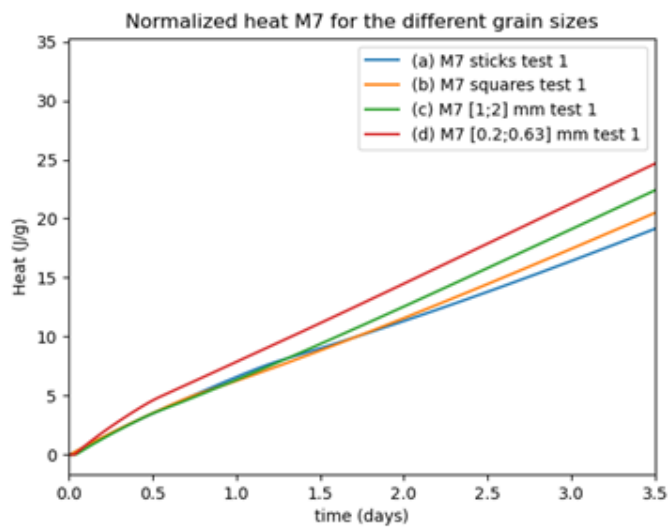


(c) Grains [1;2] mm



(d) Grains [0,2;0,63] mm

Experimental results : variation of grain size

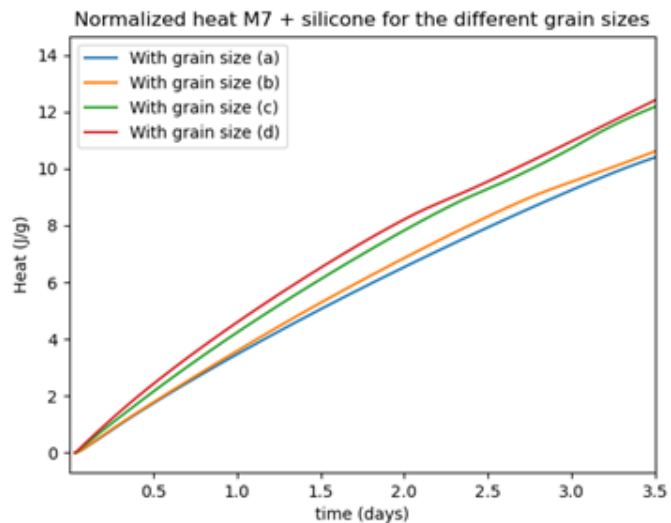


M7 with silicone

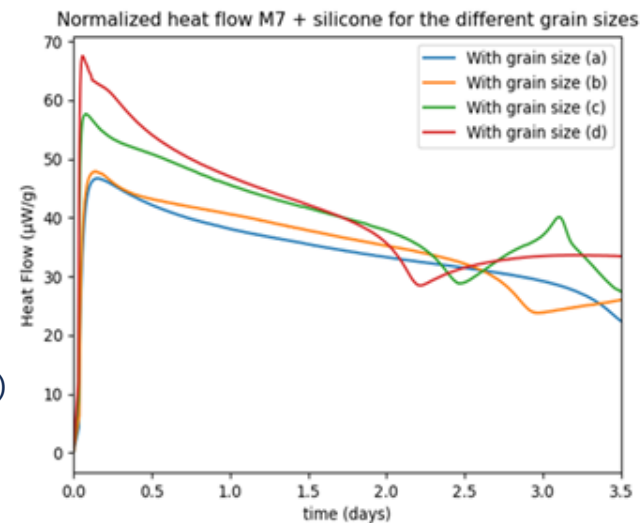
Test	Q _M (J/g)	Q _R (J/g)	Compatibility
M7 sticks + silicone	10,26	0,88	Compatible
M7 squares + silicone	10,45	0,42	Compatible
M7 [1;2] mm + silicone	12,01	0,91	Compatible
M7 [0,2;0,63] mm + silicone	12,21	0,41	Compatible

≤ 30 J/g

> 30 J/g

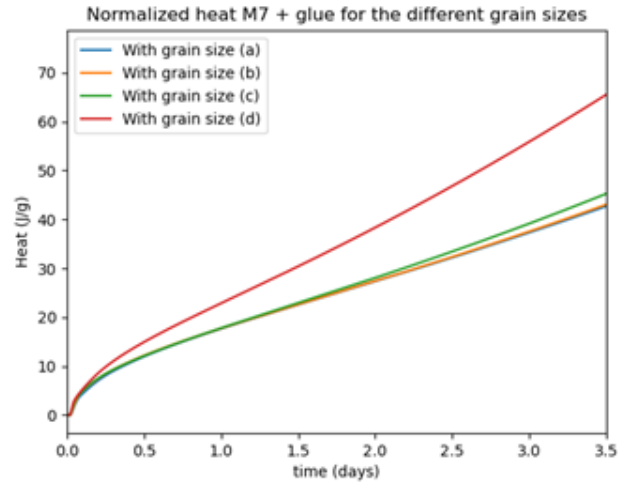


- M7 initial shape (a)
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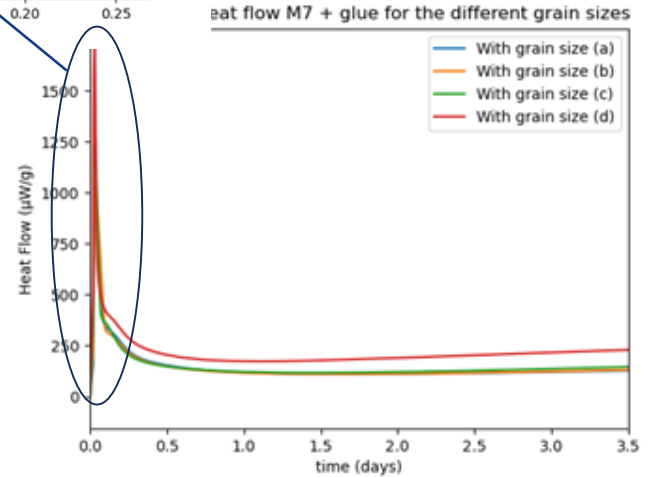
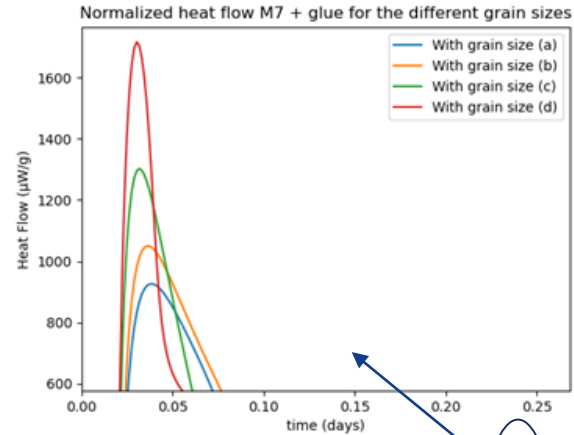


M7 with glue

Test	Q _M (J/g)	Q _R (J/g)	Compatibility
M7 sticks + glue	41,94	30,43	Incompatible
M7 squares + glue	42,26	30,22	Incompatible
M7 [1;2] mm + glue	44,39	31,37	Incompatible
M7 [0,2;0,63] mm + glue	64,16	50,24	Incompatible

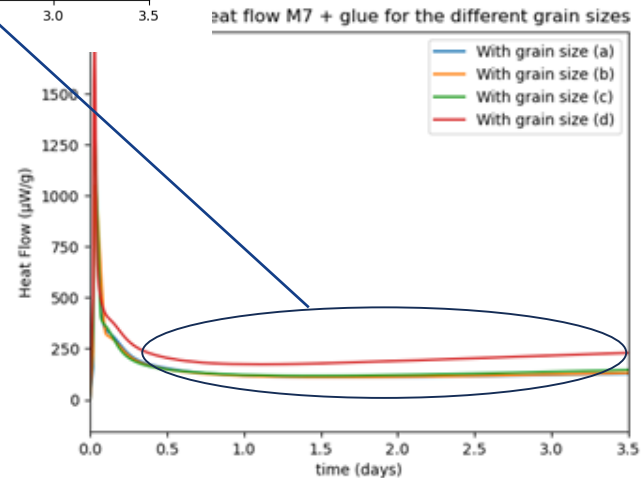
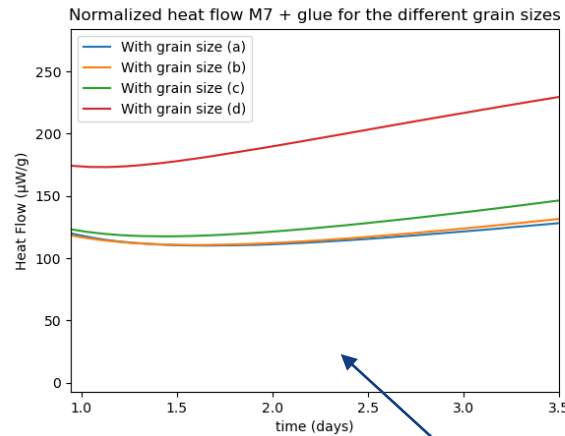
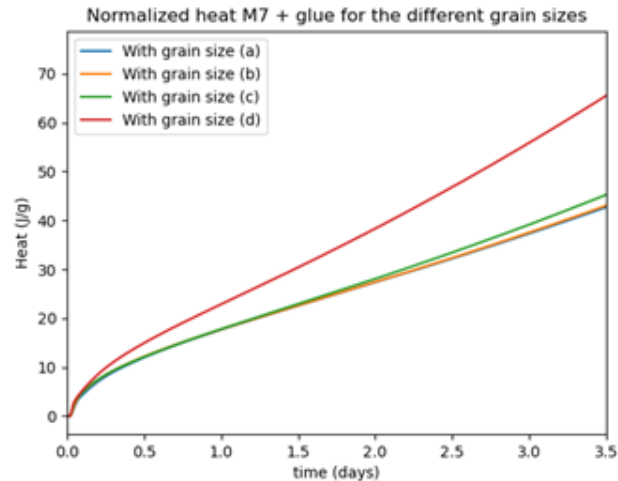


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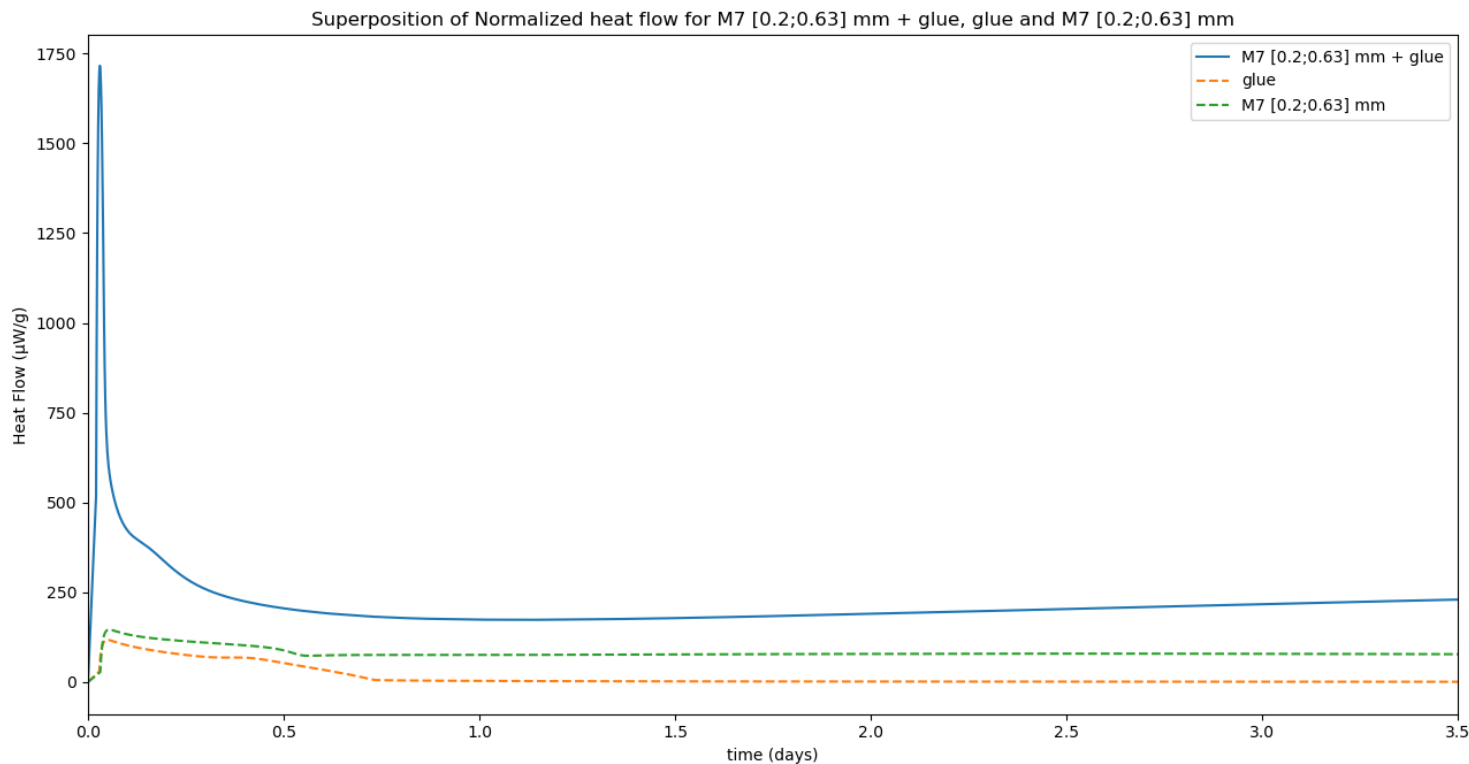


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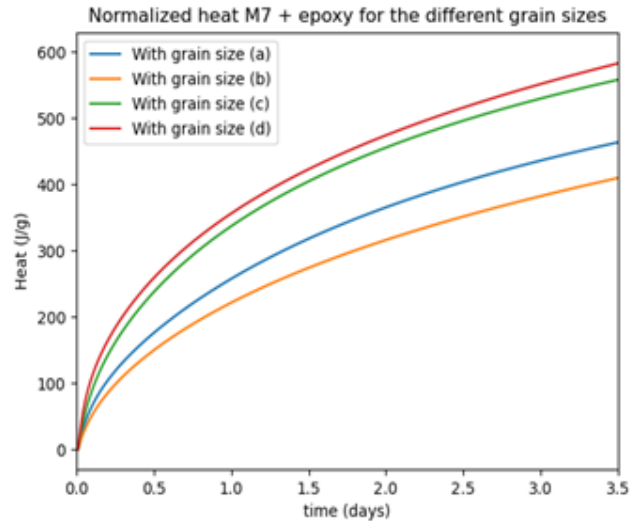


M7 with glue

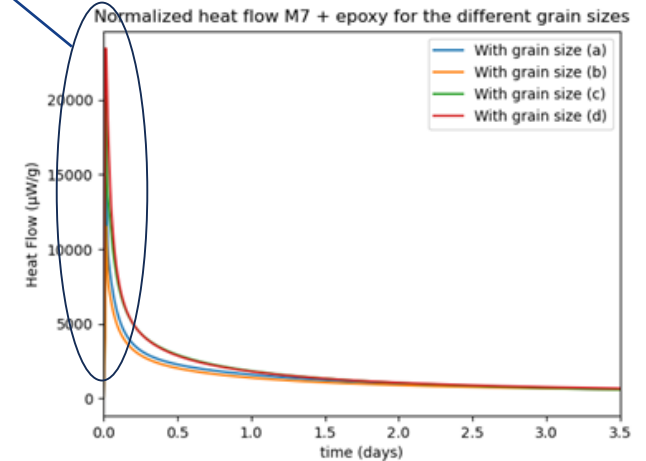
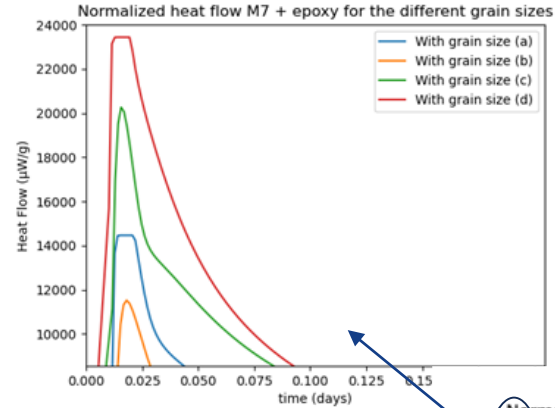


M7 with epoxy

Test	Q _M (J/g)	Q _R (J/g)	Compatibility
M7 sticks + epoxy	459,93	439,48	Incompatible
M7 squares + epoxy	406,16	387,18	Incompatible
M7 [1;2] mm + epoxy	554,21	535,37	Incompatible
M7 [0,2;0,63] mm + epoxy	578,5	558,53	Incompatible

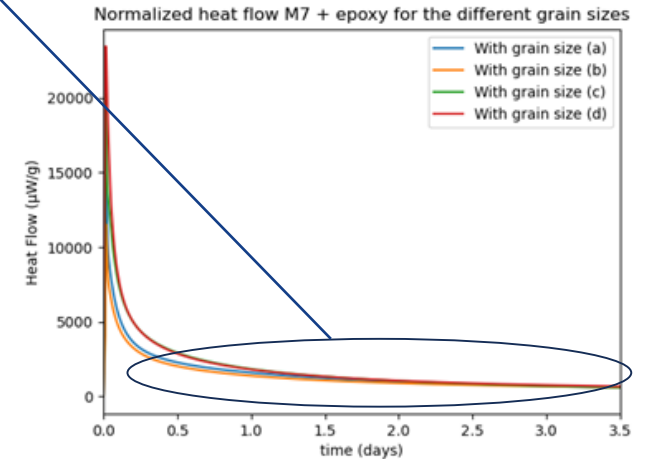
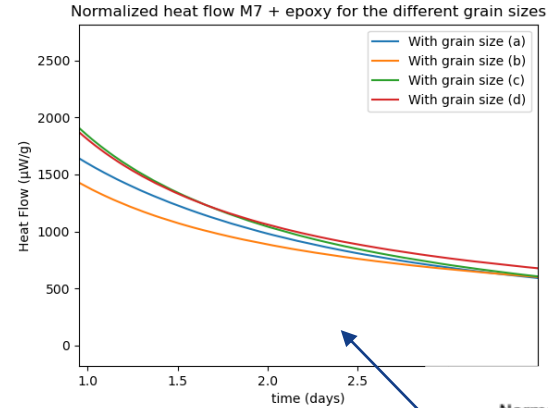
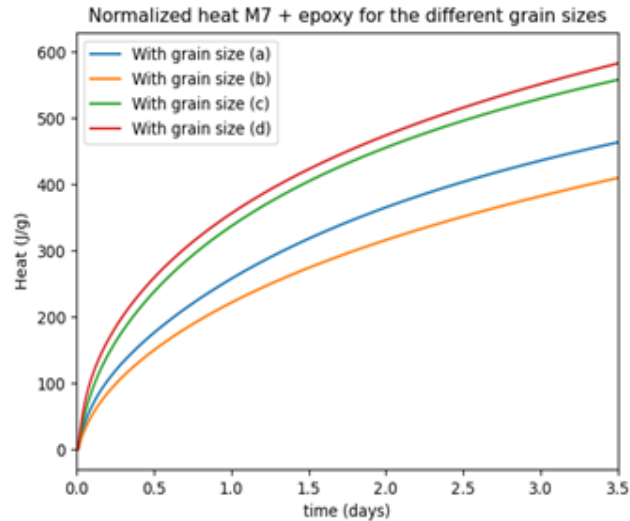


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Conclusion – What is it telling us?

- Increase the contact surface increases the rate of reaction → theory confirmed
- TGA is insufficient test to demonstrate compatibility in isolation
 - Especially for liquid nitrate ester based materials – loss / evaporation of NG
 - However is “allowable” in the STANAG
- Caution should be given to determining compatibility on the basis of single test or certain combinations;
 - eg. DSC & TGA not much better than 1 test on its own
- Effect of different grain sizes on compatibility results in HFC
 - No influence of grain size on compatibility results for compatible materials (silicone, epoxy)
 - Grain size can influence result for marginal pass materials
- Sentencing (pass/fail) criteria for HFC based on “total heat” not appropriate for material combinations where reaction is not complete.
 - May miss long terms reactions

- Many thanks go to:
 - Juliette Lo, ENSTA Bretagne
 - Cpt. Romuald Van Riet, Belgium Royal Military Academy
 - Belgium Armed Forces (supply of propellant materials)

Thank you for your attention !
Questions ?