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Inter-Batch Variation and the Effect of Casting Vacuum on Ballistic and Mechanical Properties of a High Performing Cast Composite Rocket Propellant

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ABSTRACT

A study was conducted to measure the variation in propellant properties for three consecutive batches of an identical formulation. A secondary study was performed to assess the effect of casting vacuum on propellant density, hardness, mechanical properties and burn rate. Only small differences in properties between batches were evident and most were assessed as being within the uncertainty of the measurement techniques. Batch to batch variation, as measured by percentage coefficient of variation, were below industry variances with the exception of elongation at maximum stress which was significantly higher. Casting vacuum did not significantly affect propellant hardness, density or mechanical properties. Burn rate of propellant cast without vacuum had a slightly higher burn rate and lower burn rate exponent (n). A reason for this may be that the application of casting vacuum in combination with vibration results in enhanced settling of solids including ammonium perchlorate (oxidiser) and iron oxide (ballistic modifier). A higher fraction of both solid ingredients results in a lower overall burn rate and smaller n. While casting vacuum did not adversely impact propellant properties for small plain moulds typically poured for research purposes at DSTO, casting vacuum is likely to be more important for large rocket motors with intricate grain designs. The application of casting vacuum also produced more consistent results for some properties and is recommended where possible.

RELEASE LIMITATION

Approved for public release

Inter-Batch Variation and the Effect of Casting Vacuum on Ballistic and Mechanical Properties of a High Performing Solid Composite Rocket Propellant

Executive Summary

A study was conducted to measure the variation in propellant density, hardness, mechanical properties and burn rate for three consecutive batches of an identical formulation. A secondary study was performed to assess the effect of casting vacuum on these properties. Three moulds were cast per batch with varying levels of vacuum applied: Cast directly into mould under atmospheric pressure; cast directly into mould under atmospheric pressure but with vibration and vacuum applied prior to cure; and moulds filled under vacuum with vibration and further post-fill vacuum application.

Only small differences in propellant properties between batches were evident and most were assessed as within the uncertainty of measurement techniques. Batch to batch variation, as measured by percentage coefficient of variation, were below industry variances with the exception of elongation at maximum stress which was significantly higher. This may be due to the relatively low strain capability of the propellant used for this study in relation to the uncertainty of the method employed to measure elongation at maximum stress.

Casting vacuum did not significantly affect propellant hardness, density or mechanical properties. Burn rate of propellant cast without vacuum had a slightly higher burn rate and lower burn rate exponent (n). A possible explanation for this is that the application of casting vacuum in combination with vibration results in enhanced settling of solids including ammonium perchlorate (oxidiser) and iron oxide (ballistic modifier). A higher fraction of both solid ingredients results in a lower overall burn rate and smaller n. While casting vacuum did not adversely impact propellant properties for small plain moulds typically poured for research purposes at DSTO, casting vacuum is likely to be more important for large rocket motors with intricate grain designs. The application of casting vacuum also produced more consistent results for some properties and is recommended where possible.

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Paul Smith was awarded a Bachelor of Applied Science in Chemical Technology from The University of South Australia in 1996 and initially worked in the pharmaceutical industry. In 2005 Paul completed a Bachelor of Engineering (Chemical) at The University of Adelaide with first class honours and was awarded a Doctor of Philosophy in Chemical Engineering in 2010. Paul began work at DSTO in the Weapons Propulsion group in 2009 and has been involved in research programs for high performance composite rocket propellants.

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Contents

Abbreviations

AO 2246	2,2-methylenebis (4-methyl-6-tertbutylphenol)
AP	Ammonium perchlorate
atm	Atmosphere
C/F	Coarse/fine ratio
CV	Coefficient of variation
DOA	Di-octyl adipate
EW	Equivalent weight
HTPB	Hydroxyl-terminated polybutadiene
HX-752	Trade name for an aziridine-based bonding agent
ICRPG	Interagency Chemical Rocket Propulsion Group
IPDI	Isophorone diisocyanate
NCO/OH	Isocyanate/hydroxyl ratio
P	Pressure
P_I/P_o	Plasticiser/polymer ratio
STANAG	NATO abbreviation for Standardization Agreement
T	Temperature
TPB	Triphenyl bismuth

Nomenclature

Symbol	Description	Units
a	Burn rate coefficient	$mm \cdot s^{-1} \cdot P^{-1}$
E	Young's modulus	MPa
ipm	Inches per minute	$in \cdot min^{-1}$
I_{SP}	Specific impulse	s
n	Pressure exponent	-
P	Pressure	MPa
r	Burn rate	$mm \cdot s^{-1}$
ρ	Density	$g \cdot cm^{-3}$
ε_m	Maximum strain	%
ε_b	Strain at break	%
π_k	Temperature sensitivity of pressure	$\% \cdot {}^{o}C^{-1}$
σ_m	Maximum stress	MPa

DSTO-TR-3069

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1. Introduction

Inter-batch variation and the resulting propellant property variation can have a significant impact on consistency and predictability of rocket motor performance, to the extent that missions may be jeopardised [1]. Perceptible, unacceptable variations in burn rate and mechanical properties for propellants with identical ingredients processed in identical processes have been reported [2]. Research in this area has been hampered by the complexity resulting from a large number of variables that characterise propellant ingredients, batch preparation, processing and end-use. Ramohalli [2] chose to vary final mixing time (once all ingredients had been added) while holding all other parameters constant. The influence on initial tensile modulus and mean burn rate was studied for a hydroxyl-terminated polybutadiene (HTPB), ammonium perchlorate (AP), aluminium (Al) composite rocket propellant formulation. A model was proposed based on the attrition of coarse AP caused by the shear force applied by the rotating blades of a vertical planetary action mixer. A clear trend towards increased burn rate and tensile modulus was demonstrated for longer mixing times. The trend was consistent with theoretical calculations based upon the probability of coarse AP particle comminution by rotating mixer blades.

Perez, et al. [1] analysed 71 batches (60 of which were 150 gallon mix size) of composite rocket propellant and applied a model developed for filled polymers to establish the important mixing and casting parameters that influence propellant properties. They found that propellant mechanical properties including yield and failure stress, as well as elongation but excluding hardness, showed a strong correlation with mixing time and end of cast viscosity. Propellant burn rate demonstrated no correlation with any of the measured parameters except for pre-batch storage time. Several mixes were stored for 1, 2 or 3 days at $60^{\circ}C$ after addition of all ingredients except for the curative. The effect of storage was to improve solid particle wetting and as a consequence lower viscosity. No difference in propellant mechanical properties was recorded but a significant increase in burn rate with storage time was recorded. Another finding was that a value of mixing time exists which when exceeded, constitutes optimum mixing, that is, variations in propellant properties after that point are small in comparison to earlier times. They also found that variables associated with liquid ingredient additions had no impact on propellant properties.

An important secondary consideration is the repeatability of the methods of analysis of propellant properties such as burn rate and mechanical properties. Liu [3] made an attempt at correlating the uncertainties of composite propellant strand burner test results by studying more than 3500 sets of data from the author's own resources and those available from the open literature. A model was constructed that correlated burn rate standard deviation with average burn rates for both within-batch and among-batch lots. An increase in standard deviation with mean burn rate was observed for both scenarios.

Given the aforementioned uncertainties and the prospect for variant propellant properties, a study was conducted to measure the variation in propellant properties for three consecutive batches of an identical formulation. The intent was not to determine the influence of parameters of manufacture on propellant properties but to provide a simple assessment of inter-batch variation for the existing method of manufacture in the absence of typical drivers for property variance. Thus, as far as practical, ingredient properties and

mix procedures were held constant to provide a measure of natural variation of density, hardness, burn rate and mechanical properties.

A secondary study was performed within the same three batch regime to assess the effect of casting vacuum on the aforementioned propellant properties. Vacuum is generally applied during casting to remove trapped air and to reduce the likelihood of voids within the grain. It has been reported [4] that the proportion of 'bubble contamination' influences the reproducibility of burn rate results. Yamaya [5] determined that the proportion of bubble contamination should be less than 2.0 vol% for reliable burn rate results. This study will assess whether any discernable differences in propellant properties arise from different levels of casting vacuum. Three moulds were cast per batch with varying levels of vacuum applied: cast directly into mould under atmospheric pressure; cast directly into mould under atmospheric pressure but with vibration and vacuum applied prior to cure; and moulds filled under vacuum with vibration and further post-fill vacuum application.

2. Experimental

2.1 Propellant Manufacture and Specimen Preparation

The formulation used HTPB as binder cross-linked with isophorone diisocyanate (IPDI). A bimodal coarse/fine blend of ammonium perchlorate was incorporated as oxidant with 18 wt% aluminium as fuel. Iron oxide was specified as ballistic modifier and dioctyl adipate (DOA) was incorporated as plasticiser. Other minor ingredients included a antioxidant, 2,2-methylenebis(4-methyl-6-tertbutylphenol) (AO 2246), a bonding agent (HX-752) and triphenyl bismuth as cure-rate catalyst to achieve a satisfactory pot-life. The binder was formulated with a NCO/OH ratio of 0.9 and the nominal plasticiser to polymer ratio (Pl/Po) was 0.31 (Table 1). All three batches studied in this report were processed in a 2 gallon vertical, planetary action mixer. Propellant was cast into Teflon™ coated metal moulds and cured in an oven set to 60°C for 7 days to achieve a satisfactory cure.

Table 1: Composite propellant formulation for batches P0188 – P0190.

Ingredient	Weight %
HTPB R-45 HTLO	8.87
DOA	3.00
AO 2246	0.10
Triphenyl bismuth	0.0025
IPDI	0.73
HX-752	0.30
Iron oxide	0.50
Aluminium	18.00
AP (nom. 250 µm)	51.50
AP (nom. 20 µm)	17.00

Care was exercised to ensure that as far as practical, each batch was processed in an identical fashion. All ingredients were the same lot numbers, including the fine AP that was milled in-house. All three batches were manufactured using the same methodology, that is, the same order of addition of ingredients and the same mixing conditions.

Inevitably small variances in processing will occur so where possible all anomalies that may impact propellant properties were identified and recorded. Section 3.1 provides a comprehensive list of anomalies afflicting specific batches and was used to provide possible reasons for propellant property differences.

For each of the three batches, 3 Teflon™ coated moulds were filled with propellant and each mould was cast under one of three different levels of vacuum application. Moulds had the dimensions of 175 X 50 X 75 mm (length, width, height). The three levels of vacuum application are described below:

- No vacuum – scooped from the mixer bowl directly into the mould under atmospheric pressure with no vacuum or vibration,
- Part vacuum - scooped from the mixer bowl directly into mould under atmospheric pressure but vibration and vacuum applied prior to cure,
- Full vacuum - scooped from the mixer bowl into a funnel attached to a vacuum casting bell and dropping into a mould under vacuum with vibration and further post-fill vacuum application.

This facilitated an intra-batch comparison of propellant properties for the three different casting methods and an inter-batch assessment for consistency. Following cure and removal from moulds, propellant blocks were machined into suitable test pieces for mechanical property determination and burn rate measurement as detailed in sections 2.4 and 2.5, respectively. Each block yielded approximately 8 uniaxial tensile test specimens and 30 strands for burn rate determination.

2.2 Propellant Hardness

Propellant hardness was determined with a hand-held Shore A Durometer and was the initial hardness recorded approximately 2 s after application of the meter to the surface. Measurements were performed daily at approximately the same time of day as the casting operation for a 14 day period from the date of manufacture. The first hardness reading was only performed once the propellant attained a suitable hardness for measurement (2 days after manufacture) and measurements were only taken on working week days. A consideration when interpreting hardness data is that measurements performed in the first 7 days after manufacture were taken while the propellant was curing at elevated temperature (60^oC) while those taken on the following 7 days were recorded after removal from the curing oven and with the propellant at ambient temperature (approximately 20^oC). Given the thermoplastic nature of rocket propellant, the results at elevated temperature are expected to be artificially low with reference to hardness at ambient conditions. However, the data does allow for a comparison to be made between casting methods and a comparison of that variance with natural inter-batch variance.

2.3 Propellant Density

Density measurements were performed with a Micromeritics AccuPyc 1330 pycnometer using high purity helium gas. Three individual specimens obtained from uniaxial tensile test piece off-cuts, each weighing between 3.0 and 5.5 g representing all 9 variants of propellant were determined. An average of three replicates was calculated along with a standard deviation and coefficient of variation (CV).

2.4 Mechanical Property Assessment

Tensile testing of propellant in the form of 'dog bones' or uniaxial tensile test pieces cut from cast slabs of propellant was performed to assess inter and intra-batch variation. Propellant blocks were machined into approximately 12.7 mm thick sheets and then die-cut into standard Interagency Chemical Rocket Propulsion Group (ICRPG) Class A specimens. Mechanical properties of five specimens from each of the 9 propellant variants were determined at a temperature of 20ºC. Conditioning of specimens prior to testing occurred inside the environmental chamber of an Instron 5565 tensile testing machine for a minimum of 1 hour and the cross-head speed was set at 50 mm·s^{-1}.

Instron IX software converted raw data to provide values for Modulus (E), maximum stress (σ_m), maximum strain (ε_m) and strain at break (ε_b). Modulus was calculated by the software as the maximum slope of the load versus displacement curve and was displayed as 'Modulus (Automatic)'. Maximum stress reported here is the maximum 'corrected stress' as calculated by the software and takes account of strain by adjusting for the reduction in cross-sectional area due to elongation of the specimen. Strain at maximum stress and strain at break are the values recorded via an extensometer (crosshead displacement).

2.5 Burn Rate Measurement

Burn rate of propellant was determined in a Crawford type strand burning apparatus. This involved machining cured propellant blocks into strands with dimensions of 170 X 5 X 5 mm with a bandsaw. Longitudinal faces were coated (inhibited) with an epoxy resin and burned in a Crawford-type low pressure strand burner. The method of inhibition was as detailed by Smith, et al. [6]. Burn rate at various pressures ranging from 4 to 18 MPa were determined under an atmosphere of nitrogen at 20 ± 3ºC. Burn rate dependence on pressure (Eq. 1) was calculated from an average of three burn rates at each pressure.

$$r = aP^n \tag{1}$$

3. Results and Discussion

3.1 Processing Anomalies

Despite a concerted effort, some processing anomalies did occur during propellant manufacture that had the potential to impact propellant properties. For the first batch (P0188) only 2050 g of coarse AP was dispensed rather than the prescribed 2060 g, equating to a 0.5% under addition. Since this error was made on the first batch of the campaign, it was repeated for the 2 further batches, this effectively negated the inconsistency. Further batch specific anomalies follow in Table 2 and a synopsis of the processing steps follows in Table 3.

Table 2: *Anomalies during propellant manufacture and casting*

Batch Number	Date of Manufacture	Anomaly
P0188	7/11/12	Dry clumps of material on top of mixer blades following the mix period after addition of HX-752 (step 4) and fine AP (step 7).
P0189	13/11/12	Cure rate catalyst (TPB) was not added with binder in step 1 but at step 11 (curative addition).
P0190	20/11/12	Slight delay at step 4 (HX-752 addition). Material not added immediately due to difficulty breaking up clumps of HX-752.

Table 3: *Propellant batch processing ste*

Step	Action	Stirrer Speed (rpm)	Time, No Vacuum (min)	Time, with Vacuum (min)
1	Add HTPB, DOA, AO 2246, TPB	20	2	13
2	Add iron oxide	20	1	4
3	Add aluminium	20	2	13
4	Add HX-752	20	0	5
5	Add 50% of coarse AP	20	2	13
6	Add 50% of coarse AP	20	2	13
7	Add fine AP	20	2	13
8	Scrape down	-	-	-
9	Mix	30	0	45
10	Scrape down	-	-	-
11	Add IPDI	30	1	4
12	Scrape down	-	-	-
13	Mix	30	0	20

The dry clumps of material noticed during processing of batch P0188 were likely to be coarse AP since relatively large agglomerates of AP were present in the drying trays prior to dispensing material for the mix. These agglomerates were eventually broken down during the mix cycle (no inhomogeneities noticed once the propellant was sectioned into test specimens) and should have only had a minor impact on mixing efficiency and final propellant properties. The addition of cure rate catalyst at the end of the mix process for P0189 rather than at the beginning of the mix cycle had the potential to result in less than ideal distribution throughout the batch and could have affected the cure period but should not have impacted final propellant properties to a significant extent.

A delayed addition of HX-752 for batch P0190 is unlikely to have impacted propellant properties or inter-batch variation. The time span between the end of step 3 and the beginning of step 4 for P0190 was 13 minutes and 8 and 4 minutes for batches P0188 and P0189, respectively. The slightly extra amount of time that the partially prepared propellant sat in the mixing bowl is expected to have very little effect on propellant properties.

DSTO-TR-3069

3.2 Propellant Hardness

3.2.1 Inter-Batch Variation

Hardness results for 14 days following the date of manufacture were measured with a hand-held Shore A Durometer. 'Days' refers to the number of days after the date of manufacture, that is, Day 1 is the next day after date of manufacture. Since propellant was removed from the curing oven following a 7 day cure period, measurements from day 8 were performed on a propellant block at room temperature. Inter-batch variation may be inferred from a comparison of results for each of the three batches and with the same level of casting vacuum application. Figures 1, 2 and 3 represent hardness results for the no vacuum, part vacuum and full vacuum, respectively.

Irrespective of the level of casting vacuum applied, P0190 had a higher starting (day 2) hardness than the first two batches. The most plausible explanation for this is that a slightly greater quantity of cure rate catalyst may have been added to P0190. This is possible given the difficulty associated with the measurement and addition of such a small mass (88 mg). Though the quantity can be weighed reasonably accurately, addition of the total quantity in practice is difficult since a variable amount of material may remain on the surface of the vial. The order in which the catalyst is added did not seem to have a major impact since the initial hardness for P0189 was very similar to that for P0188 and the final hardness after 8 to 14 days was fairly consistent for all 3 batches.
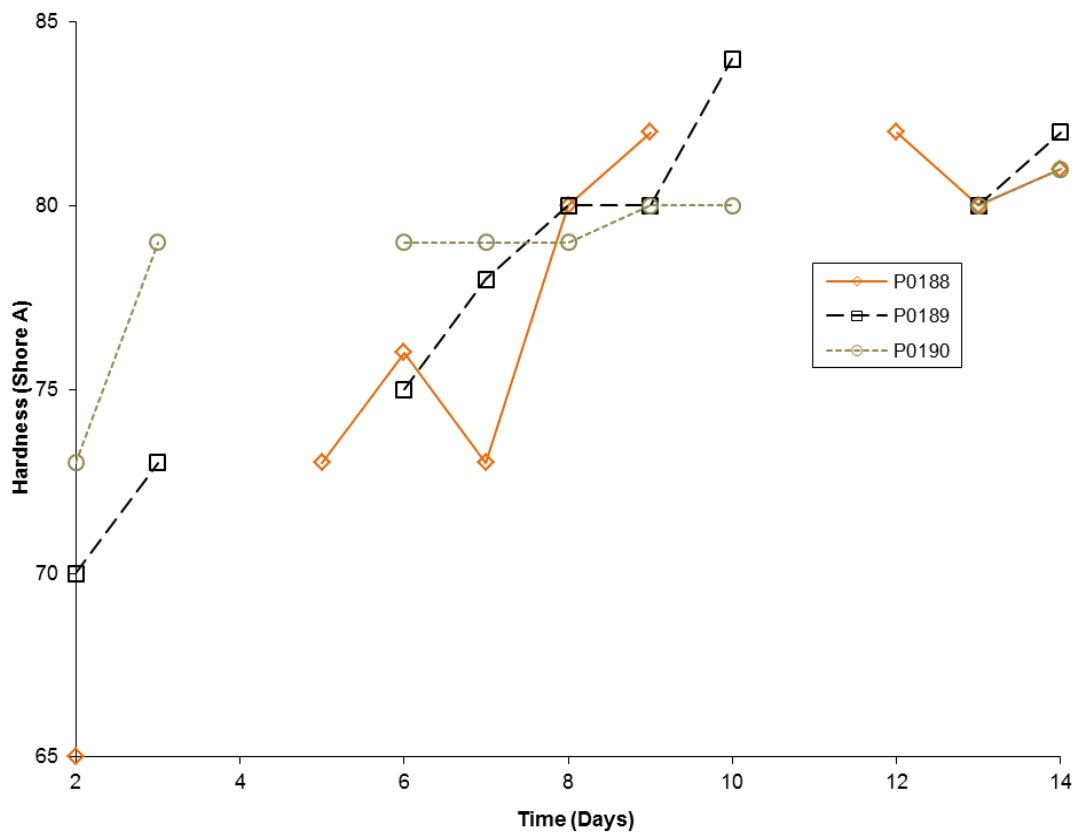

Figure 1: Propellant hardness results for batches P0188, P0189 and P0190 with no vacuum applied during casting
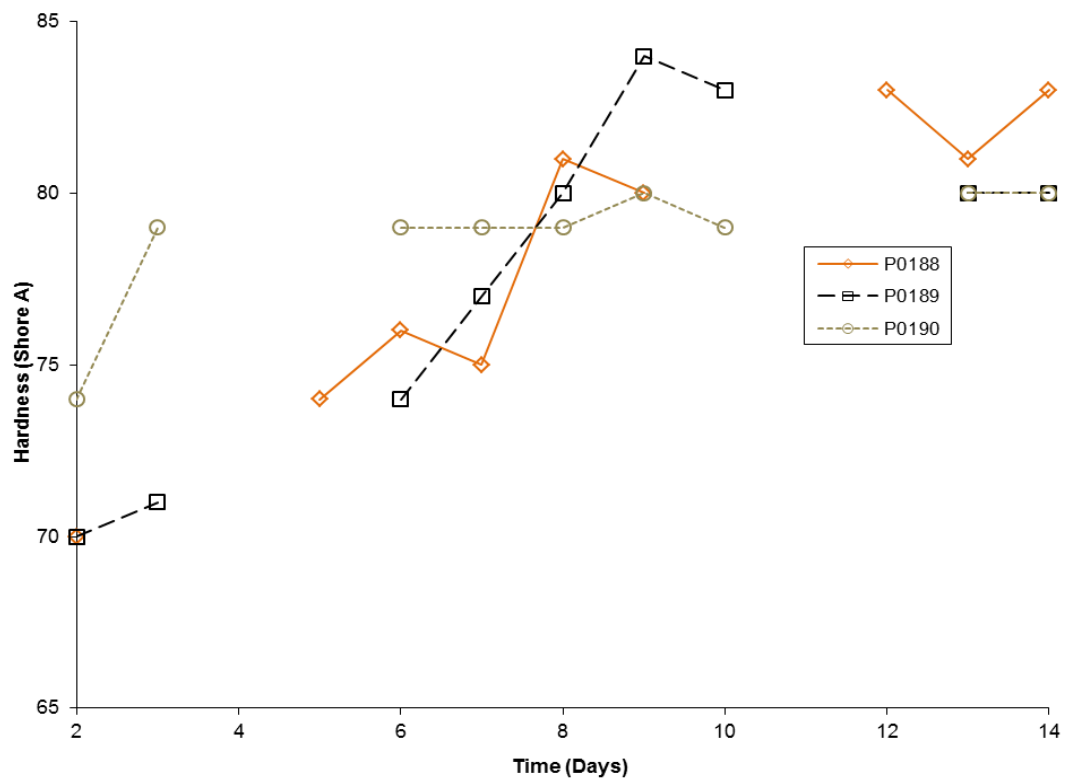
Figure 2: Propellant hardness results for batches P0188, P0189 and P0190 with partial vacuum applied during casting
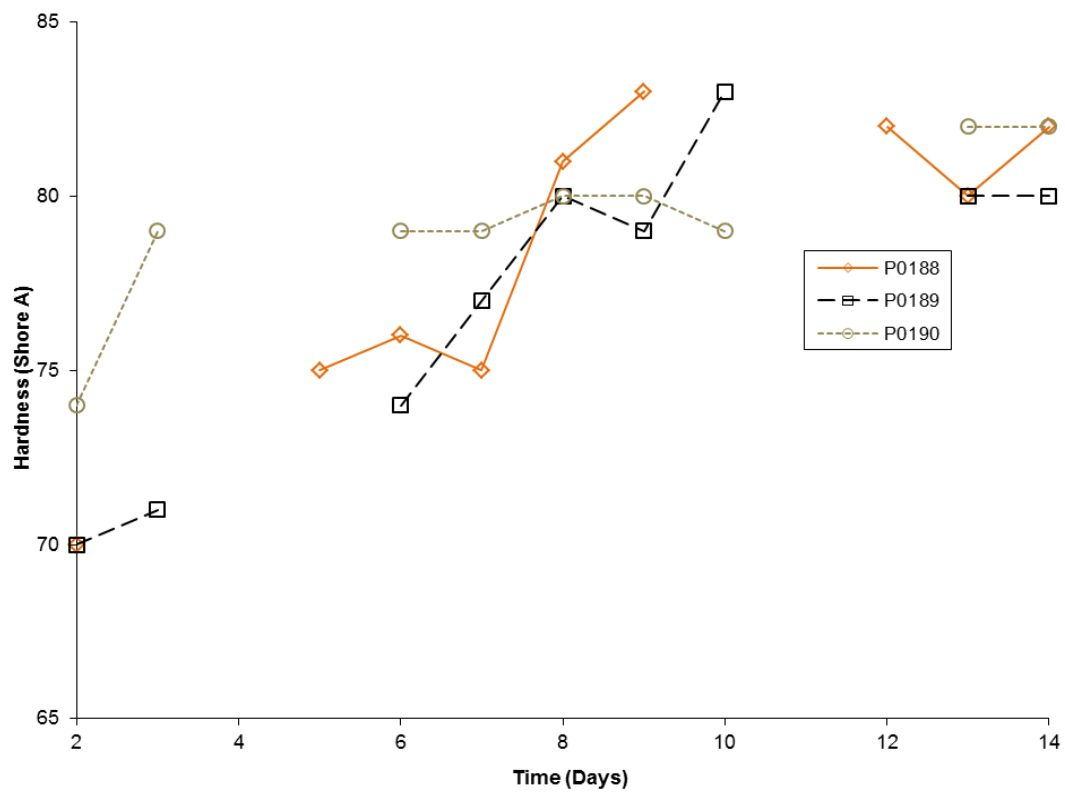

Figure 3: Propellant hardness results for batches P0188, P0189 and P0190 with full vacuum applied during casting

It is also clear that the hardness trend for P0190 is different from the first 2 batches in that it starts higher and generally does not finish as high so the trend is a much shallower one. Hardness of batches P0188 and P0189 accelerate much faster from day 6 to day 8 or 9 and only plateau after day 9 when the propellant had been out of the oven for some time and cooled completely. It is unfortunate that the true quantity of cure rate catalyst added to each batch is not known but the aforementioned trends fit with a slightly larger quantity for batch P0190.

3.2.2 Effect of Casting Vacuum

Vacuum application appears to have only a slight impact on propellant hardness (Figures 4-6). The hardness trend for P0188 is lower for no vacuum compared to partial and full vacuum but only by 2 hardness units, with the exception of the initial reading at 2 days, which was 5 units lower. For the second batch (P0189), hardness for no vacuum is generally slightly higher than for partial and full vacuum. Hardness is practically indistinguishable for the third batch with the maximum variance at 2 hardness units.

If a minor difference exists, it is for no vacuum versus some (partial or full) vacuum. That is, both partial and full vacuum application produces a slightly higher density but the difference between partial and full is indistinguishable. The overall conclusion is that hardness is not greatly affected by the application of vacuum during casting and batch to batch variation has a greater influence.
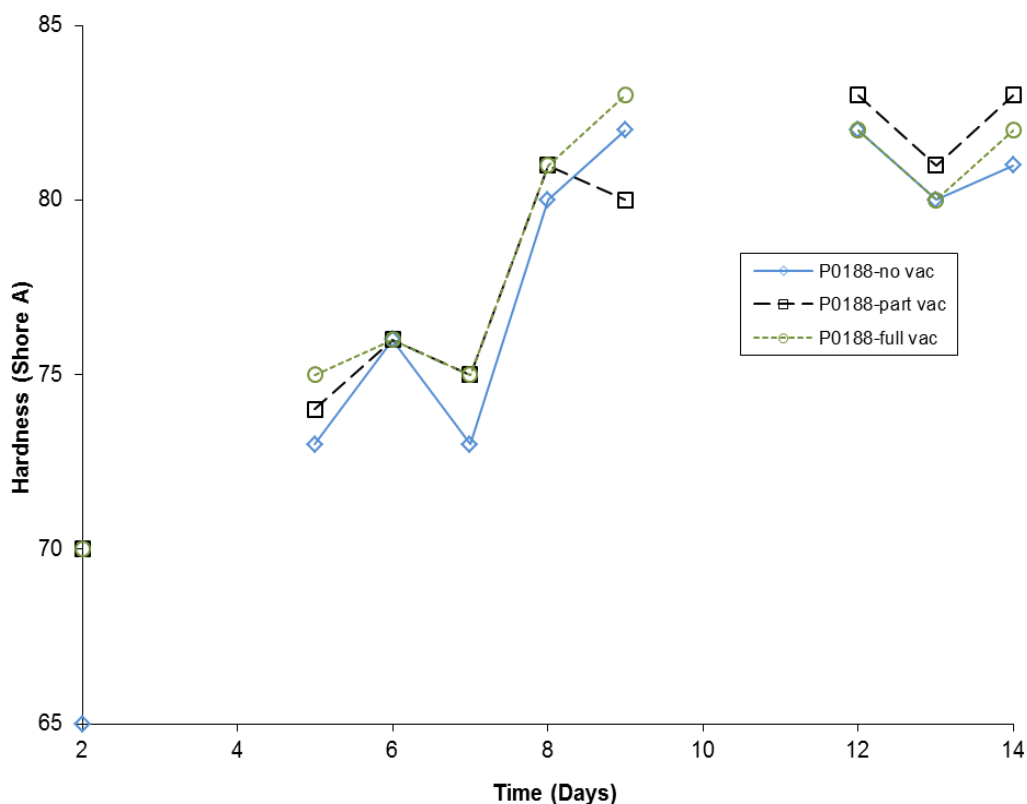

Figure 4: Propellant hardness results for batches P0188 with various levels of vacuum applied during casting
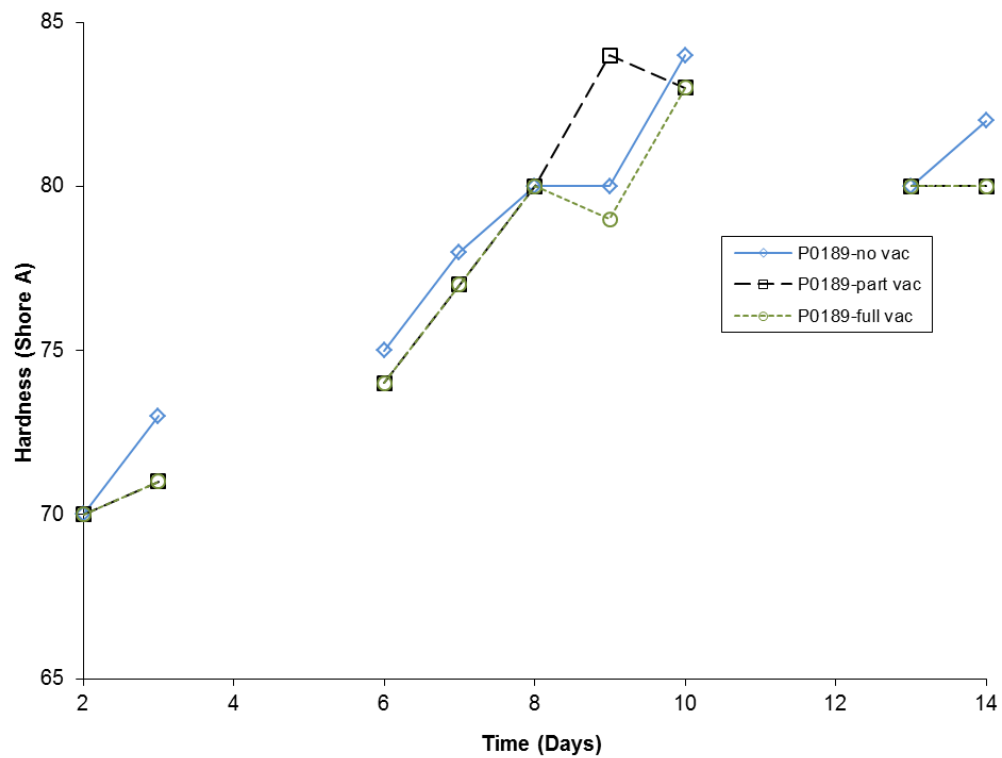
Figure 5: Propellant hardness results for batches P0189 with various levels of vacuum applied during casting
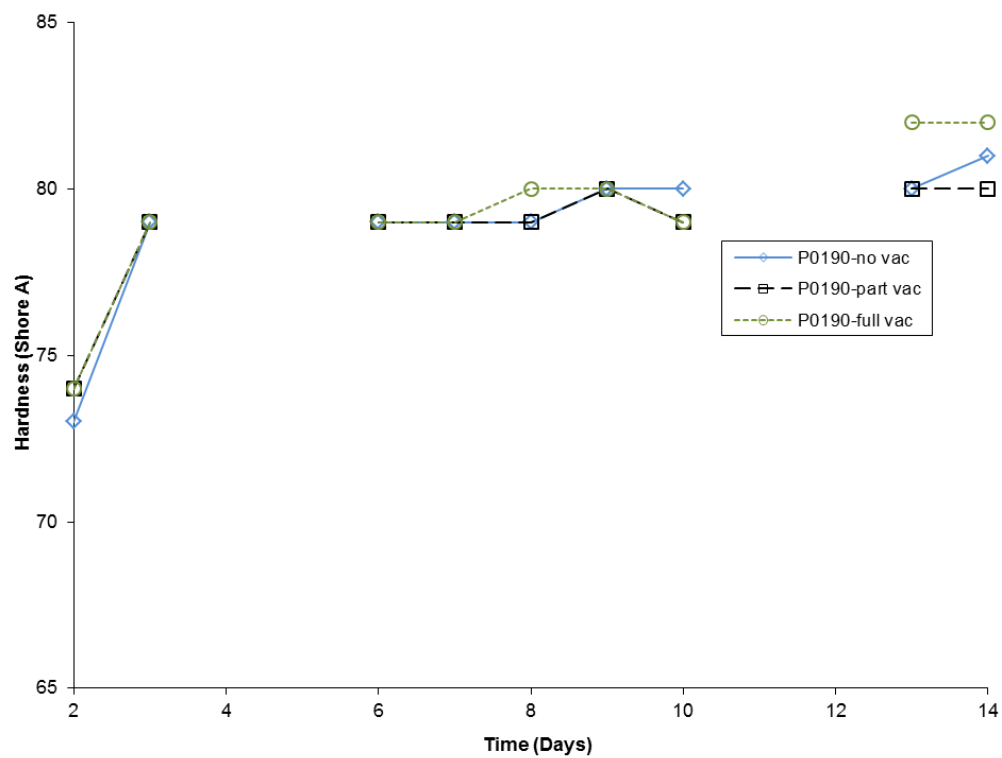

Figure 6: Propellant hardness results for batches P0190 with various levels of vacuum applied during casting

3.3 Propellant Density

3.3.1 Inter-Batch Variation

Density results for all three batches and levels of vacuum application during casting indicate only minor variances between batches for the same level of vacuum application (Tables 4-5). Table 4 in particular illustrates the variance for the 3 batches ranging from 0.0007 g·cm^{-3} for full vacuum and 0.0090 g·cm^{-3} for no vacuum, representing % CV's of 0.021 and 0.253%, respectively. However, the 'Average CV' presented in Table 5 for the three levels of vacuum application show that the uncertainty of full vacuum results is the greatest at 0.151%. This is illustrated by Figure 7 where the error bars represent average uncertainty and where a significant overlap between datasets exists. Figure 8 shows no consistent trend between vacuum application and the resultant propellant density across the three batches.

Table 4: Average densities for the three levels of vacuum application including coefficients of variation across the 3 batches (inter-batch)

Vacuum	Average Density (g·cm^{-3})	Variance (g·cm^{-3})	CV (%)
None	1.7840	0.0090	0.253
Partial	1.7848	0.0076	0.224
Full	1.7847	0.0007	0.021

Table 5: Density for the three batches and levels of vacuum application including standard deviations and coefficients of variation for the intra-batch samples

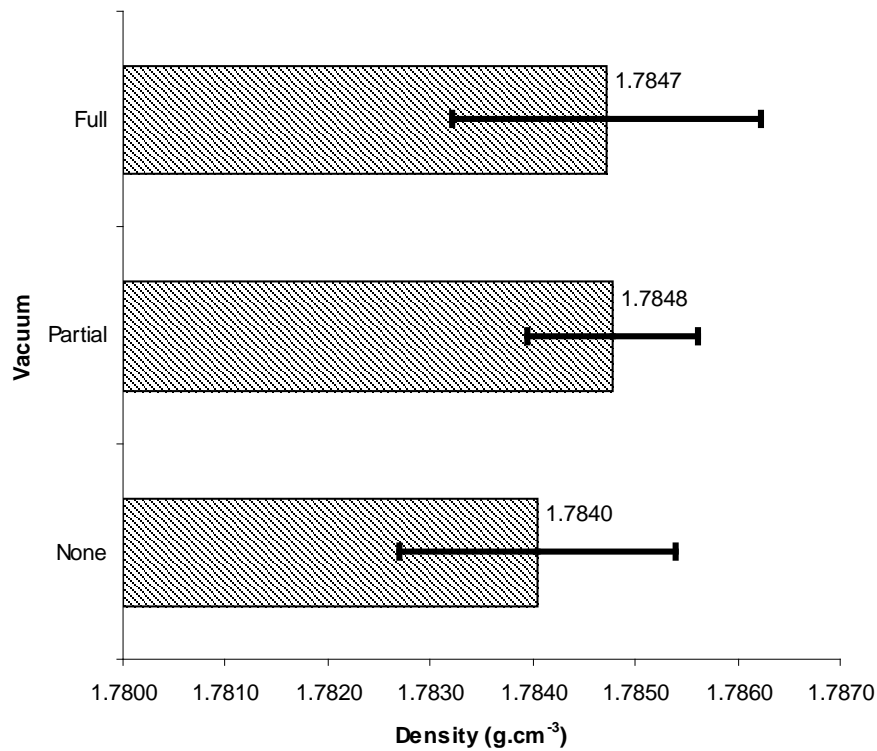| Vacuum	Batch	Density (g·cm^{-3})	SD (g·cm^{-3})	CV (%)	Average CV (%)
None	P0188	1.7846	0.00205	0.115	
None	P0189	1.7882	0.00202	0.113	0.135
None	P0190	1.7793	0.00315	0.177	
Partial	P0188	1.7817	0.00066	0.037	
Partial	P0189	1.7893	0.00152	0.085	0.083
Partial	P0190	1.7834	0.00228	0.128	
Full	P0188	1.7849	0.00211	0.118	
Full	P0189	1.7843	0.00471	0.264	0.151
Full	P0190	1.7850	0.00124	0.069	

Figure 7: Propellant density for all three batches and levels of vacuum. Error bars are for coefficients of variation for the within batch data
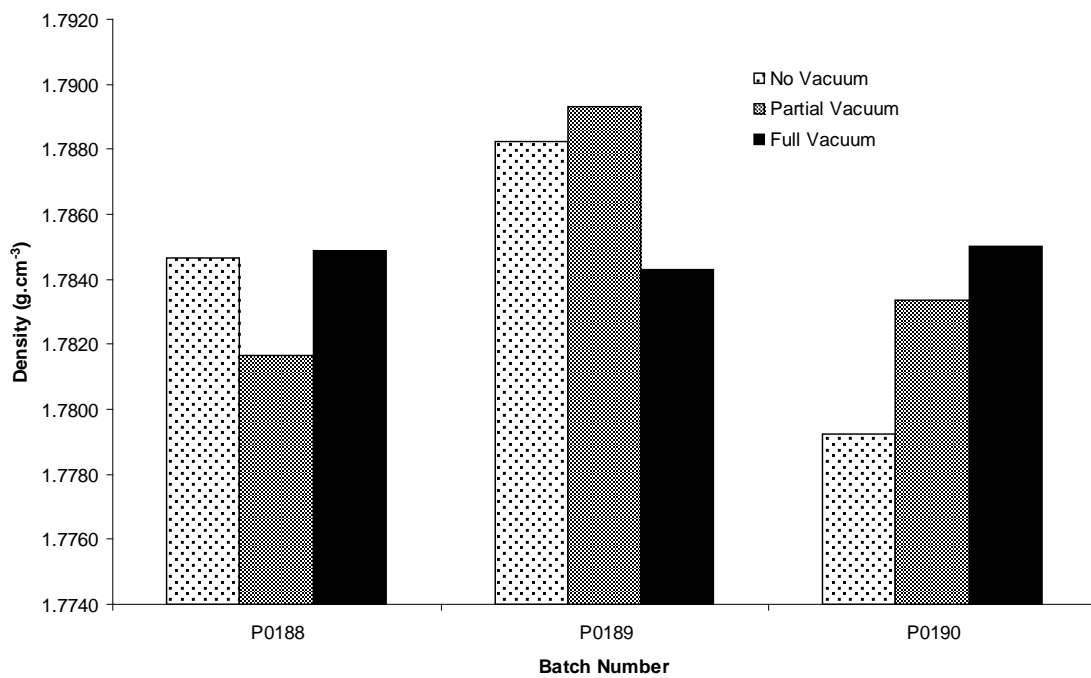

Figure 8: Propellant density for all three batches and levels of vacuum

3.3.2 Effect of Casting Vacuum

It is apparent that the absence of vacuum during casting resulted in a slightly lower density than for the case with either partial of full vacuum. Again, the results indicate that the difference between partial and full vacuum is practically indistinguishable. Given the casting process, this is not a surprising outcome. Partial vacuum entailed the filling of a relatively shallow mould (approximately 75 mm) with propellant using a spatula. The mould was then placed into a chamber where vacuum was applied for several minutes then released momentarily to collapse the voids. This occurred for a total period of approximately 15 minutes. Clearly the aforementioned process was sufficient to remove the majority of any trapped air within the propellant and to a similar extent to that for propellant cast under 'full vacuum' conditions.

Another possible effect of casting under vacuum, including during the filling of moulds (Full vacuum) is that the batch to batch consistency in terms of density results had the lowest % CV (Table 4). However, this conclusion must be seen in light of the fact that the uncertainty of the method (% CV within the 3 datasets for full vacuum) was the highest (Table 5).

3.4 Mechanical Property Assessment

3.4.1 Inter-Batch Variation

Mechanical properties including Young's Modulus, maximum stress, strain at maximum stress and strain at break were determined at 20°C for 5 specimens of each of three batches and three different vacuum applications during casting. Data presented in Tables 6-9 are average results for the 5 specimens accompanied by the standard deviation and CV and are considered intra-batch results. The average CV is simply the arithmetic average of the 3 CV's for the 3 batches within each vacuum level. Tables 10-13 contain the average value for the three different batches with the three different vacuum levels and represent inter-batch results. Variance is simply the difference between the highest and lowest of the 3 values and CV is the coefficient of variance for the three batches within each vacuum level.

Table 6: Modulus for the three batches and levels of vacuum application including intra-batch standard deviations and coefficients of variation.

Vacuum	Batch	Modulus (MPa)	SD (MPa)	CV (%)	Average CV (%)
None	P0188	25.1	0.53	2.13	
None	P0189	26.9	0.97	3.59	2.91
None	P0190	28.5	0.86	3.02	
Partial	P0188	24.8	0.84	3.37	
Partial	P0189	28.5	1.11	3.89	3.47
Partial	P0190	28.8	0.90	3.14	
Full	P0188	25.6	0.45	1.75	
Full	P0189	27.4	0.72	2.64	2.48
Full	P0190	28.6	0.88	3.06	

Table 7: Maximum stress for the three batches and levels of vacuum application including intra-batch standard deviations and coefficients of variation

Vacuum	Batch	Maximum Stress (MPa)	SD (MPa)	CV (%)	Average CV (%)
None	P0188	1.32	0.03	2.34	
None	P0189	1.31	0.05	3.84	3.41
None	P0190	1.35	0.05	4.06	
Partial	P0188	1.31	0.02	1.52	
Partial	P0189	1.40	0.03	2.17	1.84
Partial	P0190	1.34	0.02	1.84	
Full	P0188	1.32	0.02	1.86	
Full	P0189	1.36	0.02	1.28	1.61
Full	P0190	1.34	0.02	1.69	

Table 8: Strain at maximum stress for the three batches and levels of vacuum application including intra-batch standard deviations and coefficients of variation

Vacuum	Batch	Maximum Strain (%)	SD (%)	CV (%)	Average CV (%)
None	P0188	7.78	0.75	9.60	
None	P0189	7.08	0.62	8.75	9.03
None	P0190	6.93	0.61	8.74	
Partial	P0188	7.81	0.63	8.11	
Partial	P0189	7.40	0.53	7.20	5.83
Partial	P0190	6.68	0.15	2.19	
Full	P0188	8.26	0.23	2.84	
Full	P0189	7.60	0.72	9.45	5.36
Full	P0190	6.71	0.25	3.78	

Table 9: Strain at break for the three batches and levels of vacuum application including intra-batch standard deviations and coefficients of variation

Vacuum	Batch	Strain at Break (%)	SD (%)	CV (%)	Average CV (%)
None	P0188	8.56	0.85	9.91	
None	P0189	7.49	0.69	9.19	9.26
None	P0190	7.53	0.65	8.68	
Partial	P0188	8.42	0.83	9.83	
Partial	P0189	7.75	0.65	8.42	7.15
Partial	P0190	7.44	0.24	3.21	
Full	P0188	8.72	0.27	3.05	
Full	P0189	8.24	0.67	8.18	4.08
Full	P0190	7.50	0.08	1.02	

No clear inter-batch trend exists for average maximum stress within each level of vacuum application. However, there is a distinct and consistent trend for modulus, strain at maximum stress and strain at break. Within each level of vacuum application modulus is lowest for the first batch (P0188), P0189 is higher and the final batch (P0190) is the highest. For strain at maximum stress and strain at break the trend is reversed with the highest values for P0188 and lowest for P0190. Note that there is an exception with strain at break and no vacuum where P0189 is slightly lower than P0190.

The trend towards highest strain for P0188 fits with the lowest modulus for P0188 since modulus is effectively the quotient of stress over strain. Reasons for the observed trends are unclear. All raw ingredients were identical lot numbers and quantities and the batch anomalies listed in section 3.1 do not provide a possible explanation. One potential reason is that the filling techniques employed by the mix and casting personnel improved with each successive batch. Material additions and mixing times were identical for all three batches but subtle improvements in casting technique may have occurred. For example, improvements in logistics may have resulted in a reduced period between the end of the mix and the beginning of casting. This would have resulted in a reduced temperature drop in the propellant bulk and reduced progression to cure, both contributing to a lower initial viscosity leading to a more densely packed propellant. Unfortunately, the inherent uncertainty of the density method has meant that subtle differences in inter-batch density were not discernable.

3.4.1.1 Modulus

Inter-batch variation was greatest for partial vacuum with a range (variance) of 4.0 MPa and a corresponding CV of 8.14% (Table 10). Both full vacuum and no vacuum cases had variances that were similar at 3.0 and 3.4 MPa, respectively. Inter-batch variation is considered high given the magnitude of the variance in comparison with the spread of average modulus across all three vacuum applications of 0.6 MPa. Further evidence of this significance is provided by relatively low intra-batch CV's presented in Table 6 which are approximately half the inter-batch CV's for a given vacuum application. A potential reason for higher variability of the partial vacuum case may be a variable period of exposure to vacuum from batch to batch. Partial vacuum moulds were placed in the chamber sometime after filling full vacuum moulds. The length of time that the full vacuum portion received vacuum following casting was arbitrary and subject to some variation due to the perceived level of aeration of the propellant during casting. For the portion of propellant subjected to full vacuum casting, the period of time exposed to full vacuum is less significant than for the portion that only received vacuum after casting.

Table 10: *Average modulus for the three levels of vacuum application including coefficients of variation across the 3 batches*

Vacuum	Average Modulus (MPa)	Variance (MPa)	CV (%)
None	26.8	3.4	6.34
Partial	27.4	4.0	8.14
Full	27.2	3.0	5.55

3.4.1.2 Maximum Stress

Inter-batch variation of maximum stress was relatively low at 0.03 to 0.09 MPa or CV's of 1.49 to 3.39% for full and partial vacuum, respectively (Table 11). Similar low CV's were recorded for the intra-batch results reported in Table 7. Again, the CV for maximum stress was highest for the partial vacuum case which may also be attributed to variable time under vacuum as discussed in section 3.4.1.1. A variance of approximately 20-30% in terms of exposure to vacuum could exist between batches for the portion subjected to partial vacuum.

Table 11: Average maximum stress for the three levels of vacuum application including coefficients of variation across the 3 batches

Vacuum	Maximum Stress (MPa)	Variance (MPa)	CV (%)
None	1.33	0.03	1.57
Partial	1.35	0.09	3.39
Full	1.34	0.04	1.49

3.4.1.3 Strain at Maximum Stress and Strain at Break

In contrast with stress results, the inter-batch variance and corresponding CV's for strain at maximum stress and strain at break are quite large, ranging from 6.36% to 10.34% Tables 12 and 13). However, this may simply be a reflection of variability of the method since intra-batch variation is of the same order (Tables 8 and 9). A high CV is expected since actual strain values are low and potentially close to the lower bounds of resolution of the extensometer. That is, an uncertainty of 0.5 mm can have a significant effect on the results at a total extension of 6 mm whereas the % error at an extension of 30 mm is much less. This makes it very difficult to draw definitive conclusions in terms of batch to batch variation.

Table 12: Average strain at maximum stress for the three levels of vacuum application including coefficients of variation across the 3 batches

Vacuum	Maximum Strain (%)	Variance (%)	CV (%)
None	7.26	0.85	6.25
Partial	7.30	1.13	7.84
Full	7.52	1.55	10.34

Table 13: Average strain at break for the three levels of vacuum application including coefficients of variation across the 3 batches

Vacuum	Maximum Strain (%)	Variance (%)	CV (%)
None	7.86	1.07	7.72
Partial	7.87	0.98	6.36
Full	8.15	1.22	7.54

3.4.2 Effect of Casting Vacuum

Based on the average values for mechanical properties provided in Tables 10-13 some trends are evident for the three levels of casting vacuum application. Modulus is highest for partial vacuum followed closely by full vacuum and no vacuum is the lowest. This may be due to a greater void fraction (for no vacuum) and therefore less intimate contact between polymer and solid crystals resulting in premature dewetting of crystal surfaces. This is a similar phenomenon to that described by Gocmez, et al. [7] for coarse/fine ratios of AP. That is, they postulated that a greater force is required to dewet fine AP crystals due to a larger surface area/volume ratio and therefore a larger overall tensile strength. Dewetting of AP crystals from binder during the application of stress creates vacuoles which contribute to total specimen elongation. Formation of vacuoles in propellants

DSTO-TR-3069

containing a larger quantity of fine AP is less than that for propellants containing coarse particles. This contributes to a reduced overall elongation during the application of tensile stress. In this way a larger void fraction would have a similar effect to a higher coarse/fine ratio with lower tensile strength and greater elongation resulting in a lower modulus.

A similar trend is evident for maximum stress where partial vacuum had the highest stress of 1.35 MPa, full vacuum was slightly lower and no vacuum was the lowest at 1.33 MPa (Table 11). Strain at maximum stress and strain at break were highest for full vacuum and lowest for no vacuum. Both trends fit well with the void/dewetting postulate described for modulus. The significance of the greatest maximum stress and modulus for partial vacuum when full vacuum should produce the most dense, void free casting is questionable. The reason may be that, as described in section 3.3, partial vacuum may be sufficient to produce a void free grain. This is especially true for a relatively shallow mould where the sub-atmospheric pressure applied is sufficient to draw any entrapped air during pouring into the mould from the propellant mass. However, the aforementioned differences are very small any may simply be due to inherent uncertainties of the method.

3.5 Burn Rate

Burn rate of propellant was determined as detailed in section 2.5 for all 3 batches cast with full vacuum and batch P0188 no vacuum and partial vacuum to provide the data to assess inter-batch variation and the impact of vacuum on burn rate (refer to Appendix A for complete sets of data). A comparison of the pre-exponential term, exponent (n) and coefficient of determination (R^2) is summarised in the following sections.

3.5.1 Inter-Batch Variation

Data presented in Figure 9 are average results for triplicate measurements of burn rate conducted at 2 MPa increments from 4 MPa to 18 MPa. Table 14 provides a breakdown of burn rate laws across this pressure range for each of the three batches and allows for an inter-batch comparison. P0188 had a much higher CV than the other two batches resulting in a lower R^2 value. Despite a large variance for P0188, the results for all 3 batches were very similar with virtually identical values for n and similar pre-exponentials, though the value for P0188 was some distance outside the span for P0189 and P0190. The lower pre-exponential for P0188 could be dismissed as an artefact of a larger variance if it were not for the results obtained for P0188 with no vacuum and partial vacuum summarised in section 3.5.2. Those results have comparable R^2 values to those for P0189 and P0190 and with pre-exponentials that are representative of the value obtained for full vacuum. This is suggestive of a real difference in results for batch P0188.

Table 14: Burn rate law exponents, standard deviations and coefficients of variation for all 3 batches

Batch	Pre-exponential	Exponent (n)	R^2	Average SD (mm·s⁻¹)	Average CV (%)
P0188	6.669	0.289	0.934	0.221	1.64
P0189	6.716	0.285	0.971	0.128	1.05
P0190	6.755	0.287	0.967	0.125	0.95

A look at the raw results for P0188 reveals one potential outlier of 14.91 mm·s^{-1} at 10 MPa, whereas the other two values at 10 MPa were 13.37 and 13.45 mm·s^{-1}. Exclusion of that value from the results produces the comparison in Table 15. It is evident that the data set for P0188 is now representative with very similar R^2, average SD and Average CV. This also confirms the conclusion that the pre-exponential for P0188 is lower than for P0189 and P0190.

Table 15: Burn rate law exponents, standard deviations and coefficients of variation, excluding the outlier value for P0188 at 10 MPa

Batch	Pre-exponential	Exponent (*n*)	R^2	Average SD (mm·s^{-1})	Average CV (%)
P0188	6.641	0.289	0.957	0.121	0.92
P0189	6.716	0.285	0.971	0.128	1.05
P0190	6.755	0.287	0.967	0.125	0.95
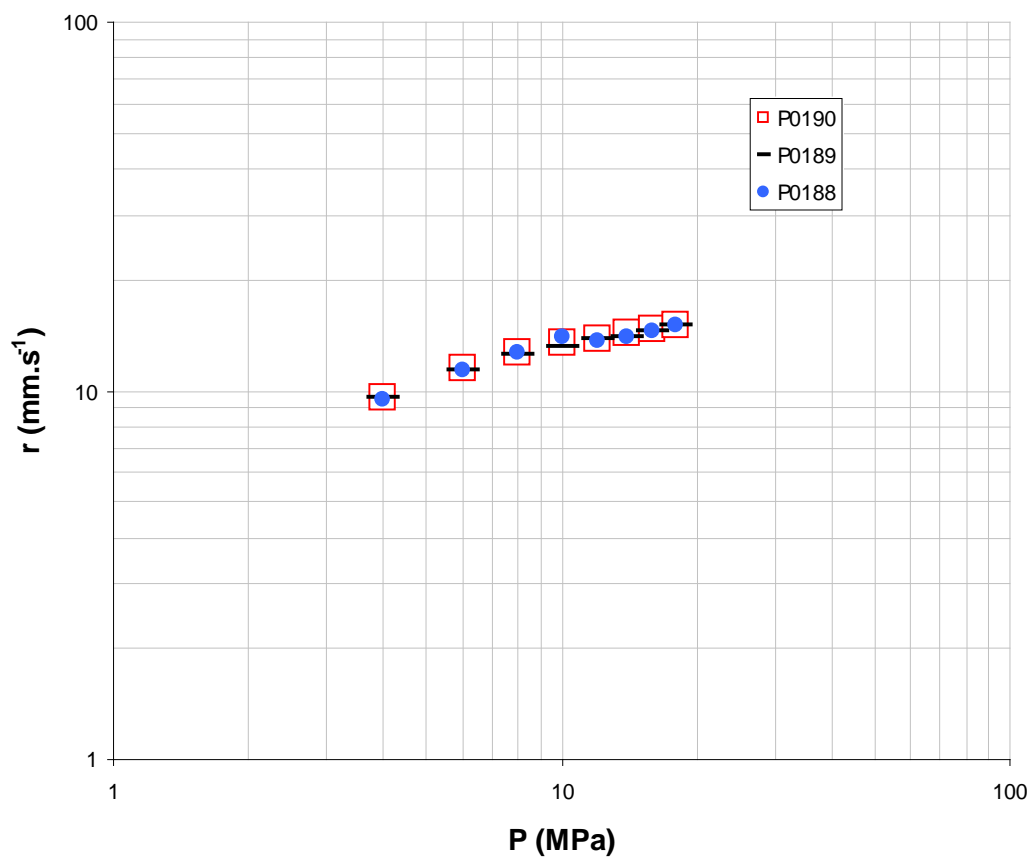
Figure 9: Propellant burn rate laws for all three batches cast with full vacuum

3.5.2 Effect of Casting Vacuum

Data presented in Figure 10 are average results for triplicate measurements of burn rate conducted at 2 MPa increments from 4 MPa to 18 MPa. Table 16 provides a breakdown of burn rate laws for each of the three batches and allows for a comparison of casting vacuum levels. A similar spread in terms of pre-exponential and R^2 is evident for the casting vacuum data and inter-batch variation data. Similar average SD and % CV are also in

DSTO-TR-3069

evidence. The case of no vacuum has however produced significant differences for both the pre-exponential and n resulting in a slightly different shape to the curve as in evidence in Figure 10. Though percentage coefficient of variation for the no vacuum case is slightly higher than for partial and full vacuum, this does not fully explain the difference in burn rate law. As suggested earlier, the difference between partial and full vacuum in terms of packing is most likely very small. A possible reason for the difference may be due to a greater degree of AP particle settling in the partial and full vacuum cases.

Settling of the relatively dense solids such as AP, Al and iron oxide commences immediately following the end of the mixing process and motion of the mixer blades. The no vacuum-case moulds were filled first resulting in the shortest time for solids settling in the bulk material in the mixing bowl. Additionally, the application of vibration in combination with vacuum would an increase in settling rate for solids within the moulds. Both factors would lead to a gradient within each mould and a relatively high solids content at the bottom of the mould. Strands were cut from the lower portion of each mould while dog bones were cut from the top of the mould. It is possible that the strands for both partial and full vacuum had a slightly higher solids content than those for no vacuum. There is also the possibility of a higher settling rate for coarser AP particles and/or the higher density iron oxide particles. It is a well-established phenomenon that composite propellant with a higher proportion of fine AP has a higher burn rate and a higher n [8]. However, a higher iron oxide content has been linked to a lower n [9].The data for no vacuum shows a larger pre-exponential (higher intrinsic burn rate) and smaller n.

Table 16: *Burn rate law exponents, standard deviations and coefficients of variation for batch P0188 and all 3 levels of applied casting vacuum*

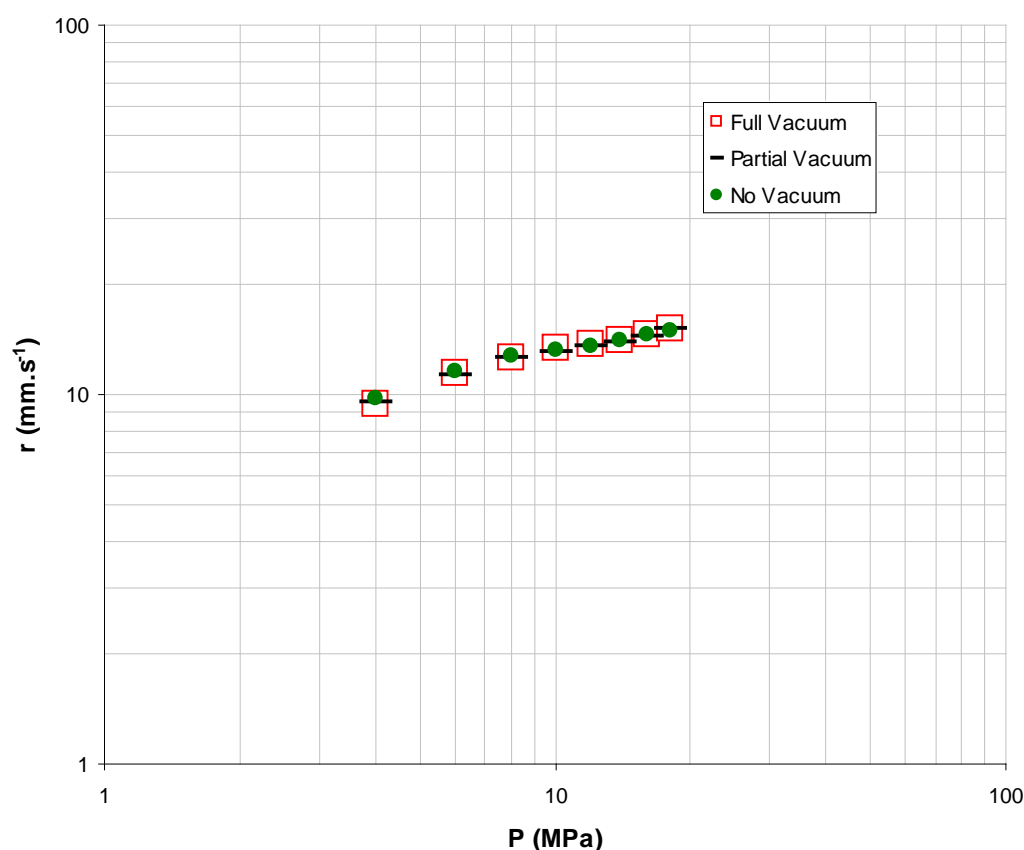| Batch	Pre-exponential	Exponent (n)	R^2	Average SD (mm·s^{-1})	Average CV (%)
No Vacuum	7.039	0.264	0.965	0.129	1.02
Partial Vacuum	6.664	0.285	0.972	0.105	0.82
Full Vacuum	6.641	0.289	0.957	0.121	0.92

Figure 10: Propellant burn rate laws for all three levels of applied casting vacuum

3.6 Summary

It must be emphasised that in all cases any differences in average values are small and the overall conclusion must be that batch to batch variation is small and the impact of casting vacuum level is also small. Perez, et al. [1] surveyed 60, 150 gallon propellant mixes and found % CV's for burn rate of 3.0%, 5.4% for yield stress, 7.7% for yield elongation and 3.5% for hardness (Table 17). The data presented here (Smith), with the exception of elongation, are well below the variance observed by Perez et al.

Table 17: Propellant property variations (% CV) as reported by Perez, et al. [1] and as recorded by this author. The hardness value for Smith was taken at day 7, the burn rate value is a % CV at 8 MPa. Smith (vacuum) results are for batch P0188.

Author	Burn rate	Yield stress	Yield Elongation	Hardness	Average CV (%)
Smith	0.8	1.5	10.3	2.6	3.8
Perez, et al.	3.0	5.4	7.7	3.5	4.9
Smith (vacuum)	0.6	0.4	3.4	1.6	1.5

Coefficient of variation values for the effect of casting vacuum (Smith (vacuum)) are significantly lower than those reported by Perez, et al. and much lower than those for inter-batch variation. This adds further weight to the argument that casting vacuum has little impact on propellant properties including hardness, density, burn rate and

mechanical properties. A word of caution however, is that casting vacuum did not have a significant impact with the small, open shapes cast here. That is, this was a stylised exercise with relatively shallow, rectangular moulds. The casting of complex shapes and with long vertical path lengths such as large motors with star shaped grains may be much more sensitive to the application of casting vacuum. The work presented here demonstrates that the simple laboratory scale casting conducted at DSTO is not particularly sensitive to casting vacuum. However, there is some evidence that either partial or full vacuum results in more consistent propellant properties. It is therefore recommended that vacuum casting be continued as a practice at DSTO where circumstances allow.

Another cautionary note is that the data presented by Perez, et al. was for a 150 gallon mixer whereas the data presented here is for a 2 gallon mixer. A scaling factor exists where larger mixers impart a lower shear due to increased blade clearances, resulting in higher end of mix viscosities for larger mixers [10]. However, Perez, et al. found that mixer size had little impact as their 1 gallon mixes produced similar results to those for the 150 gallon mixer when corrected for differences in end of mix viscosity.

Liu [3] Surveyed burn rate results from 2885 batches and developed a relationship for standard deviation for intra-batch results with a R^2 of 0.86 (Eq. 2).

$$\text{Average SD} = 0.0428 * \text{average burn rate } (mm \cdot s^{-1}) - 0.121 \qquad (2)$$

Applying this equation to the average burn rate for batch P0190 with full vacuum, a % CV of 3.4% is produced. That value matches very well with the data of Perez, et al. but is much higher than the values obtained in this work. This suggests that the batch to batch variation achieved at DSTO is lower than that achieved in industry. The results presented here provide re-assurance that DSTO's practices are sufficiently tight to facilitate the elucidation of changes in propellant properties as a result of deliberate changes in formulation and/or processing.

4. Conclusions

The mixing and casting of three cast composite rocket propellant batches at DSTO using the same lot numbers of ingredients and the same mixing conditions produced results for hardness, density, mechanical properties and burn rate that were practically identical. Only small differences in properties between batches were evident and most were assessed as within the uncertainty of measurement techniques. Batch to batch variation as measured by percentage coefficient of variation were below industry variances with the exception of elongation at maximum stress which was significantly higher. This may be due to the relatively low strain capability of the propellant used for this study in relation to the uncertainty of the method employed to measure elongation at maximum stress.

Casting vacuum did not significantly affect propellant hardness, density or mechanical properties. Burn rate of propellant cast without vacuum had a slightly higher burn rate and lower burn rate exponent (*n*). A possible explanation for this is that the application of casting vacuum in combination with vibration results in enhanced settling of solids including ammonium perchlorate (oxidiser) and iron oxide (ballistic modifier). A higher

fraction of both solid ingredients results in a lower overall burn rate and smaller n. While casting vacuum did not adversely impact propellant properties for small plain moulds typically poured for research purposes at DSTO, casting vacuum is likely to be more important for large rocket motors with intricate grain designs. The application of casting vacuum also produced more consistent results for some properties and is recommended where possible.

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Appendix A: Propellant Strand Burn Results

Table A1: Low pressure strand burn results for P0188, full vacuum coated with epoxy R180/H180

Pressure (MPa)	Burn Time (s)	Burn Rate (mm·s^{-1})	Average Burn Rate (mm·s^{-1})	Standard Deviation (mm·s^{-1})
4	13.60	9.34		
4	13.35	9.51	9.44	0.09
4	13.42	9.46		
6	11.05	11.49		
6	11.24	11.30	11.40	0.10
6	11.14	11.40		
8	10.04	12.65		
8	10.12	12.55	12.63	0.08
8	10.10	12.70		
10	9.50	13.37		
10	8.52	14.91	13.91	0.86
10	9.44	13.45		
12	9.23	13.76		
12	9.36	13.57	13.65	0.10
12	9.33	13.61		
14	9.02	14.08		
14	9.03	14.06	13.95	0.21
14	9.26	13.71		
16	8.63	14.72		
16	8.86	14.33	14.53	0.19
16	8.74	14.53		
18	8.47	14.99		
18	8.33	15.25	15.08	0.15
18	8.47	14.99		

Table A2: *Low pressure strand burn results for P0189, full vacuum coated with epoxy R180/H180*

Pressure (MPa)	Burn Time (s)	Burn Rate (mm·s^{-1})	Average Burn Rate (mm·s^{-1})	Standard Deviation (mm·s^{-1})
4	13.09	9.70		
4	13.13	9.67	9.57	0.21
4	13.61	9.33		
6	11.07	11.47		
6	10.90	11.65	11.44	0.23
6	11.35	11.19		
8	10.09	12.59		
8	10.19	12.46	12.53	0.06
8	10.13	12.54		
10	9.54	13.31		
10	9.60	13.23	13.24	0.07
10	9.64	13.17		
12	9.20	13.80		
12	9.23	13.76	13.77	0.03
12	9.24	13.74		
14	9.18	13.83		
14	9.20	13.80	13.95	0.22
14	8.94	14.21		
16	8.68	14.63		
16	8.66	14.67	14.59	0.11
16	8.78	14.46		
18	8.45	15.03		
18	8.43	15.07	15.10	0.10
18	8.35	15.21		

Table A3: *Low pressure strand burn results for P0190, full vacuum coated with epoxy R180/H180*

Pressure (MPa)	Burn Time (s)	Burn Rate (mm·s^{-1})	Average Burn Rate (mm·s^{-1})	Standard Deviation (mm·s^{-1})
4	13.21	9.61		
4	13.08	9.71	9.64	0.06
4	13.24	9.59		
6	11.06	11.48		
6	10.94	11.61	11.47	0.15
6	11.23	11.31		
8	9.89	12.84		
8	9.92	12.80	12.74	0.14
8	10.10	12.57		
10	9.56	13.28		
10	9.44	13.45	13.45	0.16
10	9.33	13.61		
12	9.12	13.93		
12	9.11	13.94	13.89	0.08
12	9.21	13.79		
14	8.78	14.46		
14	8.86	14.33	14.32	0.15
14	8.96	14.17		
16	8.65	14.68		
16	8.64	14.70	14.75	0.10
16	8.54	14.87		
18	8.33	15.25		
18	8.48	14.98	15.08	0.14
18	8.45	15.03		

Table A4: *Low pressure strand burn results for P0188, no vacuum coated with epoxy R180/H180*

Pressure (MPa)	Burn Time (s)	Burn Rate (mm·s^{-1})	Average Burn Rate (mm·s^{-1})	Standard Deviation (mm·s^{-1})
4	13.26	9.58		
4	12.97	9.79	9.74	0.14
4	12.91	9.84		
6	10.79	11.77		
6	11.03	11.51	11.57	0.18
6	11.11	11.43		
8	10.02	12.67		
8	9.94	12.78	12.68	0.09
8	10.08	12.60		
10	9.69	13.11		
10	9.72	13.07	13.18	0.17
10	9.49	13.38		
12	9.36	13.57		
12	9.48	13.40	13.54	0.13
12	9.30	13.66		
14	9.03	14.06		
14	9.12	13.93	13.97	0.08
14	9.13	13.91		
16	8.77	14.48		
16	8.84	14.37	14.47	0.10
16	8.72	14.56		
18	8.49	14.96		
18	8.60	14.77	14.92	0.14
18	8.45	15.03		

Table A5: *Low pressure strand burn results for P0188, partial vacuum coated with epoxy R180/H180*

Pressure (MPa)	Burn Time (s)	Burn Rate (mm·s^{-1})	Average Burn Rate (mm·s^{-1})	Standard Deviation (mm·s^{-1})
4	13.44	9.45		
4	13.29	9.56	9.50	0.05
4	13.39	9.48		
6	11.22	11.32		
6	11.42	11.12	11.24	0.10
6	11.26	11.28		
8	10.09	12.59		
8	10.07	12.61	12.54	0.11
8	10.23	12.41		
10	9.92	12.80		
10	9.56	13.28	13.04	0.24
10	9.74	13.04		
12	9.42	13.48		
12	9.42	13.48	13.55	0.12
12	9.28	13.69		
14	9.12	13.93		
14	9.14	13.89	13.90	0.03
14	9.16	13.86		
16	8.84	14.37		
16	8.78	14.46	14.42	0.05
16	8.80	14.43		
18	8.50	14.94		
18	8.54	14.87	14.98	0.14
18	8.39	15.14		

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19. ABSTRACT
A study was conducted to measure the variation in propellant properties for three consecutive batches of an identical formulation. A secondary study was performed to assess the effect of casting vacuum on propellant density, hardness, mechanical properties and burn rate. Only small differences in properties between batches were evident and most were assessed as being within the uncertainty of the measurement techniques. Batch to batch variation, as measured by percentage coefficient of variation, were below industry variances with the exception of elongation at maximum stress which was significantly higher. Casting vacuum did not significantly affect propellant hardness, density or mechanical properties. Burn rate of propellant cast without vacuum had a slightly higher burn rate and lower burn rate exponent (n). A reason for this may be that the application of casting vacuum in combination with vibration results in enhanced settling of solids including ammonium perchlorate (oxidiser) and iron oxide (ballistic modifier). A higher fraction of both solid ingredients results in a lower overall burn rate and smaller n. While casting vacuum did not adversely impact propellant properties for small plain moulds typically poured for research purposes at DSTO, casting vacuum is likely to be more important for large rocket motors with intricate grain designs. The application of casting vacuum also produced more consistent results for some properties and is recommended where possible.