

Understanding the Influence of Manufacturing and Material Parameters on the Mechanical Properties of Polymer-Clay Composites: An Exploratory Statistical Analysis

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Abstract. Polymers are used in various industrial applications due to their ease of production, light weight and ductility. Fillers such as clays are added to polymers to improve a range of factors such as material processing, thermal properties, fire retardance and cost. However, adding clays may negatively impact the mechanical performance, affecting the strength, stiffness and impact resistance of the composite. In addition, manufacturing parameters (e.g. number of extrusions, press time, sample cooling method) may also have an influence on the resulting composite system.

This paper analyses a set of previously obtained experimental results on various clay fillers added to high density polyethylene (HDPE). The composite material was compounded using an extrusion process and manufactured into tensile test samples by means of hot pressing. Various manufacturing parameters (number of extrusions, press time, sample cooling method), material (polymer grade, clay type, clay weight loading) and testing parameters (strain rate) were investigated to determine their influence on the mechanical properties of the composite system. The results showed wide variability, necessitating a statistical approach.

Exploratory data analysis and statistical analysis techniques are applied to the historical tensile test data to gain insight not only into the influence on, but the relationships and interactions with the various parameters of interest with regard to the mechanical properties, specifically strength (ultimate tensile strength) and ductility (percentage elongation at failure). The statistical analysis concluded that all parameters, except for press time, have a statistically significant influence on the mechanical properties of interest. This influence was then qualified through the insights gained from the exploratory data analysis, combined with the knowledge of the material and manufacturing process. The conclusions and insights gained from these analyses will be used to inform a statistical design of experiments to quantify the manufacturing variability and reliability in future work.

INTRODUCTION

Polymer-clay composites have received considerable attention in recent years, both in research and industry, as they allow for the manipulation of the base polymer material to improve on the thermo-mechanical properties by adding small micro- or nano-sized clay fillers (platelets, fibers or particles) [1, 2, 3, 4, 5, 6, 7]. By adding these clay fillers the final composite material may have improved material, thermal and electrical properties, as well as a high strength to weight ratio at lower costs, all desirable traits for various industrial applications [1, 2, 4, 6, 7, 8]. However, even when clay is added with the intention of improving a certain set of properties for a specific application area, we still need to understand the effect on the mechanical properties. A number of authors have indicated that by adding a small amount of filler, generally less than or equal to 5 wt%, there is an improvement in the mechanical properties compared to a neat polymer [2, 3, 5, 6, 8]. However, our previous work has shown that when the filler content exceeds 10 wt% the material properties begin to degrade [9]. The influence of the polymer-clay material system (e.g. clay and polymer type, clay loading, dispersion and the interaction between the clay and polymer) on the mechanical properties have been the subject of much research [3, 4, 5, 8, 10].

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In addition to the material system, the manufacturing process has an influence on the composite morphology and consequently the thermo-mechanical properties [6, 7, 8]. This is largely due to the change in polymer-clay morphology (i.e. type of dispersion, polymer chain formation, crystallinity, ductility, etc.) which is dependent on the manufacturing procedure [6, 8]. Albdiry *et al.* [8] compared various experimental studies, each using different manufacturing methods, and concluded that the mechanical properties of the resulting polymer-clay composites are affected by the manufacturing method. On the other hand, Tarapow *et al.* [11] investigated the influence of mixing parameters (mixing speed and time) while mixing a polymer and clay with an intensive mixer they developed. Their study concluded that the mixing parameters had no significant effect on the clay dispersion or the mechanical properties. Gaining more insight and understanding into the effects of the manufacturing process on the mechanical properties of the polymer-clay composite will allow for improved composite design practices [8].

The aim of this study is therefore to gain insight into the effect of various manufacturing and composite material parameters on the mechanical properties. From 2016 to 2018 a number of experimental studies were conducted each considering different material, manufacturing and testing conditions. Before conducting any future experimental studies it is necessary to get an overall sense of the previously obtained experimental data. This will be done by first investigating and understanding the historical data set to identify any main characteristics, patterns or anomalies. This process is referred to as an exploratory data analysis (EDA). Once we have a better understanding of the data set a statistical analysis will be conducted to quantify the effects observed.

MATERIALS AND METHODS

Melt-mixing combined with extrusion is a simple, cost-effective method for producing thermoplastic polymers while also providing sufficient clay dispersion [2, 5, 6, 8]. The polymer-clay composite systems in this study were compounded using a twin-screw extruder before applying a compression moulding technique to manufacture the tensile test samples [12, 13, 14, 15]. The composite material, manufacturing and testing system parameters considered in this study are summarised in Table I. The total number of samples includes five specimens for each case investigated as per the requirements of ASTM D638-02 to ensure statistical significance [16].

The experimental data considered in this study was collected from 2016 to 2018 as part of a larger study to better understand the composite material system and manufacturing procedure and how the mechanical properties are affected by these. This was done by focussing on different aspects of the system as part of the final year undergraduate mechanical engineering research projects. In 2016, the aim was to investigate the influence of the number of extrusion passes and clay loading on HDPE A7260 and Alcamizer 1 [12]. With the number of extrusions fixed, in 2017 the aim was to find the mechanism which causes changes in the mechanical properties by considering different polymer grades, clay types, clay loading and varying the vertex hot press time [13]. Following this, in 2018 the number of extrusions and press time was fixed along with the clay type and polymer grade. Two research projects were defined, the first investigated the effects of higher clay loadings and again considered the influence of the number of extrusions at these higher loads [14] and the second would investigate the effects of the sample cooling method and the strain rate [15]. A summary of these experimental studies is provided in Table I. Clay loading as a design variable is the only commonality between the experimental studies from 2016 to 2018, with the exception of the second study in 2018 which only considered neat HDPE. Strain rate, press time, number of extrusions and sample cooling method are constant variables in at least three of the four experimental studies.

Materials, Manufacturing and Testing

One of the most versatile and widely used thermoplastics is polyethylene because of its toughness, near-zero moisture absorption, chemical inertness, low coefficient of friction, ease of processing and electrical properties [7, 17]. High density polyethylene (HDPE) was chosen for the polymer matrix, with different grades considered during the experimental study. The polymer was supplied in pellet form. To better understand the effects of the clay filler three fillers were considered, (1) Alcamizer 1, which is not compatible with HDPE, (2) DHT4-A which is compatible with HDPE and (3) an uncoated Alcamizer 1 to determine the effect of surface coating which was obtained by removing surfactant using a solvent.

To manufacture the testing samples the HDPE is pulverised into a fine powder and mixed with the required clay loading in a bag for 45 min to ensure dispersion [6]. The polymer-clay mixture is then extruded into a long wire using a TK28P CFAM twin screw extruder after which the wire is fed through a chipper to obtain pellets. This process was

TABLE I: Summary of the experimental studies conducted indicating the design and constant variables considered.

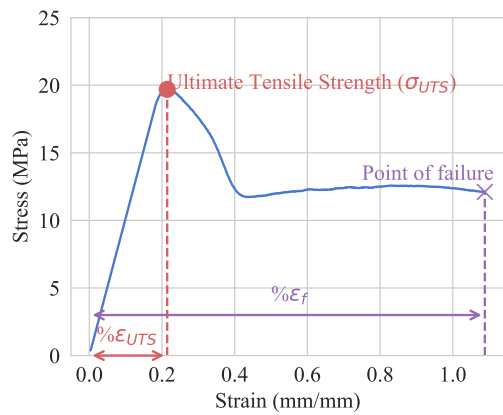
Year	Experiment	Design Variable		Constant Variable		Total Samples
2016	Influence of the number of extrusions	Clay loading	0, 5, 10 wt%	Press Time	20 min	79
		Nr of extrusions	1, 2, 3	Sample Cooling	Air	
				Strain Rate	5 mm/min	
				Clay Type	Alcamizer	
				Polymer Type	HDPE A7260	
2017	Influence of the material system and press time	Polymer Type	HPDE B7750, C7260, D7255	Nr of extrusions	2	538
		Clay Type	Alcamizer, DHT4A, Uncoated Alcamizer	Sample Cooling	Air	
		Clay loading	0, 2.5, 5, 7.5 wt%	Strain Rate	5 mm/min	
		Press Time	25, 30, 35, 45 min			
2018	Influence of extrusions and higher clay loadings	Clay loading	10, 15, 20 wt%	Press Time	25 min	20
		Nr of extrusions	1, 2	Sample Cooling	Air	
				Strain Rate	5 mm/min	
				Clay Type	DHT4-A	
				Polymer Type	HDPE B7750	
2018	Influence of sample cooling method and strain rate	Sample Cooling	Air, Quenched, Furnace	Polymer Type	C7260	65
		Strain Rate	5, 100, 500 mm/min	Clay Type	None	
				Clay loading	0 wt%	
				Nr. of Extrusions	2	
				Press Time	25 min	

repeated a second time when two extrusions were required and a third time for three extrusions. The heating zone temperatures for the extruder were set to 105 °C (heating zone 1), 165 °C (heating zone 2), 195 °C (heating zone 3) and 185 °C (die heating zone).

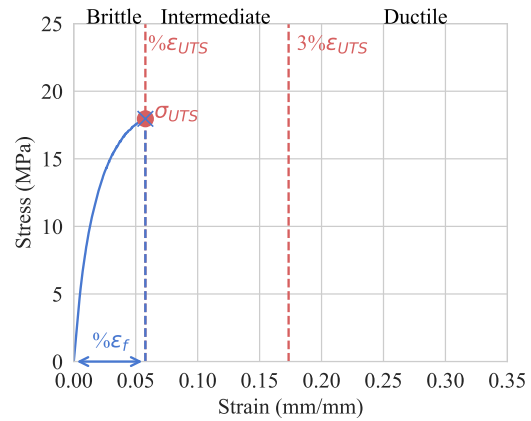
To create the tensile testing samples the polymer nanocomposite pellets were stacked into specimen moulds designed and manufactured by Parschau [12] based on the ASTM D638-02 Type I [16] dimensions. The specimens were then compressed using a vertex hot press at a fixed temperature of 180 °C and a pressure of 15 MPa applied in increments for the total press time of 25, 30, 35 or 45min. The pressure increments were applied from 0 to 7.5 MPa and held for 30 sec before increasing it to 15 MPa for 5 min. The pressure is then dropped back to 0 MPa where it is increased to 7.5 MPa and held for 30 sec before increasing it to 15 MPa and holding for the remainder of the press time. The pressure is released back to 0 MPa before removing the mould. Finally specimens were cured using one of three sample cooling methods, (1) air cooled at room temperature by placing the mould in the laboratory until the mould was cool enough to touch, (2) quenched by placing the mould immediately after removal from the press into cold water or (3) furnace by placing the moulds into a furnace which was heated to 60 °C before switching it off. Once the moulds were cooled, the samples were removed and the surfaces finished, using a carpenters knife and fine grade sanding paper, to remove any excess material or impurities due to the pressing procedure that could influence the tensile testing results. To obtain the desired mechanical properties a tensile test was conducted using a Lloyd Instruments LRX Plus 5kN Tensile Machine considering displacement rates of 5, 100 and 500 mm/min as described in Table I.

Data Processing

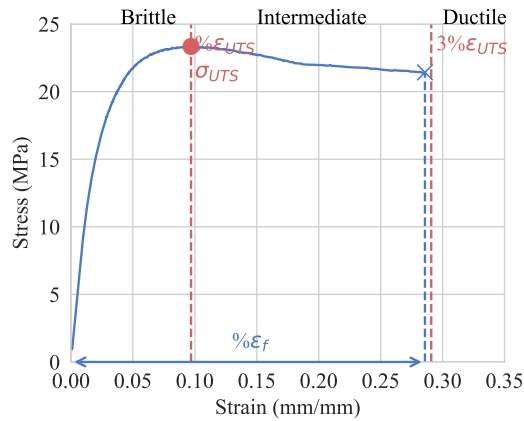
Raw data files were obtained from the tensile testing for all cases considered, except for the 2016 data investigating the number of extrusions. The data for that study was digitized from the report of Parschau [12] as the raw data files were no longer available. Tensile testing was conducted to failure and a clip gauge was used, however digitized data was only available up to the Ultimate Tensile Stress (σ_{UTS}). The raw tensile data files for the experimental tests conducted in 2017 and 2018 were processed correcting for potential sample slippage, machine compliance and the linear elastic region as discussed in ASTM D638-02 [16]. In 2017 a clip gauge was used and the tensile test was conducted to a predefined strain of approximately 30%. In 2018 no clip gauges were used as it kept slipping, and tensile tests were conducted until failure occurred. The mechanical properties of interest are then determined from the processed



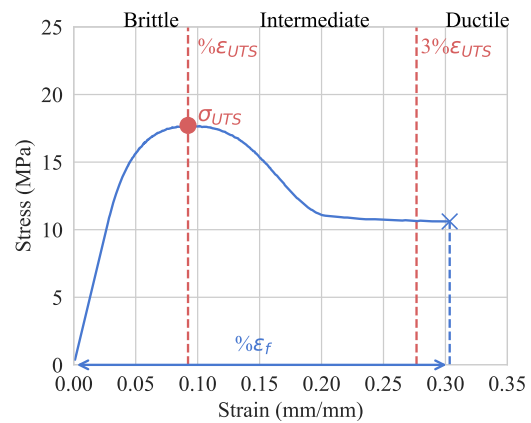
(a) Defining variables (HDPE B7750/10 wt% DHT4A)



(b) Brittle Failure (HDPE B7750/2.5 wt% Alcamizer)



(c) Intermediate Failure (HDPE B7750/2.5 wt% Alcamizer)



(d) Ductile Failure (HDPE B7750/2.5 wt% Alcamizer)

FIGURE 1: Stress-strain curve defining the different variables and failure types in this study.

stress-strain graphs as shown in Fig. 1a where the Ultimate Tensile Stress (σ_{UTS}) is defined as the maximum stress the material can achieve, the percentage elongation at the Ultimate Tensile Stress ($\% \epsilon_{UTS}$) is the strain measured at σ_{UTS} and the percentage elongation to failure ($\% \epsilon_f$) is the strain measured at the recorded point of failure.

As the tensile testing conditions for each of the experimental studies were different as mentioned in the previous paragraph, $\% \epsilon_f$ is merely considered to give an indication of the material ductility and potential material property degradation. For this reason the failure mode was grouped into three categories based on the value of $\% \epsilon_f$ in relation to $\% \epsilon_{UTS}$:

- Brittle:** if $0 < \% \epsilon_f \leq \% \epsilon_{UTS}$
- Intermediate:** if $\% \epsilon_{UTS} < \% \epsilon_f \leq 3\% \epsilon_{UTS}$
- Ductile:** if $\% \epsilon_f > 3\% \epsilon_{UTS}$

The defined grouping criteria above is visually demonstrated in Fig. 1b for a Brittle failure, Fig. 1c for an Intermediate failure and Fig. 1d for a Ductile failure. In this study when a composite material system undergoes primarily brittle type failures as the clay loading is increased the material properties are said to degrade.

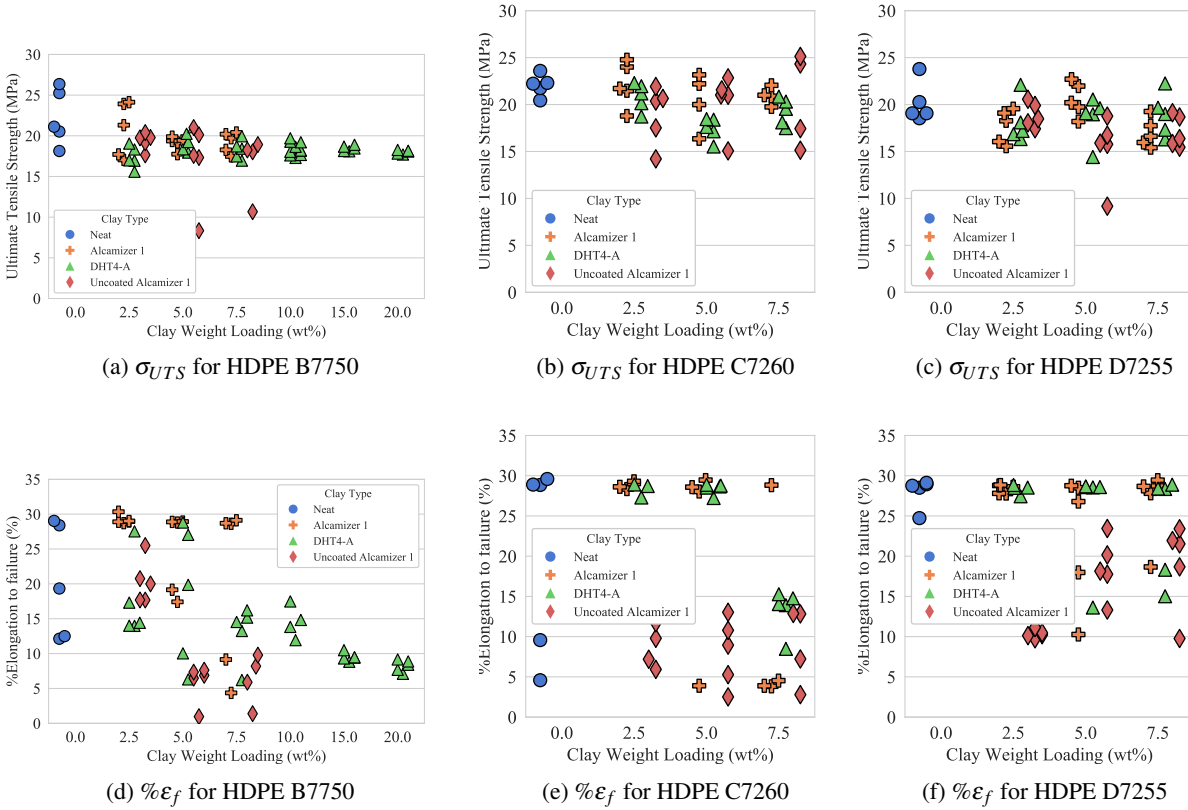


FIGURE 2: Influence of the Material System variables on σ_{UTS} (top row) and $\% \epsilon_f$ (bottom row). The marker for the neat HDPE is a blue circle, Alcaminizer 1 an orange plus, DHT4-A a green triangle and Uncoated Alcaminizer 1 a red diamond.

EXPLORATORY DATA ANALYSIS

The exploratory data analysis investigates the effect of the different manufacturing (number of extrusions, press time and sample cooling method), testing (strain rate) and material parameters (polymer grade, clay type and clay weight loading) on the mechanical properties of interest, i.e. σ_{UTS} and $\% \epsilon_f$.

According to literature this study expects to observe an increase in σ_{UTS} if there is a good interaction between the filler and polymer [4, 5, 6] and a decrease if the interaction between the filler and polymer is poor [2, 4]. HDPE is a semi-crystalline polymer [17] and it has been reported that in general with the addition of clay particles a decrease in $\% \epsilon_f$ is observed regardless of the interaction between the clay and polymer [2].

Table II provides the summary statistics of the σ_{UTS} and $\% \epsilon_f$ responses for the different combinations of the experimental variables. The number of observations for the different combinations of experimental conditions is very different, which indicates that the experiments were not performed according to a statistical experimental design i.e., the experimental conditions are unbalanced. Table II also provides the mean, standard deviation and the standard error of the mean for each of the experimental conditions.

Material System Influence

The influence of the material system variables, discussed in Table I, on the mechanical properties of interest (i.e. σ_{UTS} and $\% \epsilon_f$) is shown in Fig. 2.

A general observation is that as clay is added to the polymer system, there is a decrease in the σ_{UTS} from the

TABLE II: Summary statistics of the σ_{UTS} and $\% \epsilon_f$ responses for the different combinations of the experimental variables

Clay Loading	Strain Rate	Extrusions	Clay Type	Polymer Grade	Nr. of Observations	Mean		Standard Deviation		Standard Error	
						σ_{UTS}	$\% \epsilon_f$	σ_{UTS}	$\% \epsilon_f$	σ_{UTS}	$\% \epsilon_f$
0	5	1	Neat	A7260	7	18.80	21.40	1.53	3.42	0.58	1.29
0	5	2	Neat	A7260	11	21.54	21.68	2.81	3.68	0.85	1.11
0	5	2	Neat	B7750	23	22.26	20.24	2.68	9.71	0.56	2.02
0	5	2	Neat	C7260	55	20.38	146.96	4.22	121.04	0.57	16.32
0	5	2	Neat	D7255	20	19.61	26.18	2.76	5.80	0.62	1.30
0	5	3	Neat	A7260	10	21.72	21.52	3.09	6.16	0.98	1.95
0	100	2	Neat	C7260	15	26.41	50.55	3.32	29.15	0.86	7.53
0	500	2	Neat	C7260	15	31.73	18.58	1.24	1.86	0.32	0.48
2.5	5	2	Alcamizer 1	B7750	20	19.96	27.06	2.30	6.02	0.51	1.35
2.5	5	2	Alcamizer 1	C7260	20	22.65	26.29	1.98	6.65	0.44	1.49
2.5	5	2	Alcamizer 1	D7255	20	18.14	24.14	1.56	7.91	0.35	1.77
2.5	5	2	DHT4-A	B7750	10	18.71	18.84	1.92	4.75	0.61	1.50
2.5	5	2	DHT4-A	C7260	10	20.50	23.25	1.19	9.12	0.38	2.88
2.5	5	2	DHT4-A	D7255	10	17.96	26.72	2.08	5.08	0.66	1.61
2.5	5	2	Uncoated Alcamizer 1	B7750	10	19.62	18.88	2.21	3.61	0.70	1.14
2.5	5	2	Uncoated Alcamizer 1	C7260	10	20.17	9.82	2.92	4.04	0.92	1.28
2.5	5	2	Uncoated Alcamizer 1	D7255	10	18.16	13.03	1.56	4.33	0.49	1.37
5	5	1	Alcamizer 1	A7260	8	22.28	22.62	0.60	4.72	0.21	1.67
5	5	2	Alcamizer 1	A7260	9	22.69	21.47	0.80	2.83	0.27	0.94
5	5	2	Alcamizer 1	B7750	20	19.45	22.19	1.98	8.99	0.44	2.01
5	5	2	Alcamizer 1	C7260	20	20.56	25.52	1.68	7.86	0.37	1.76
5	5	2	Alcamizer 1	D7255	20	19.50	21.99	2.47	8.66	0.55	1.94
5	5	2	DHT4-A	B7750	10	19.14	23.58	1.82	8.63	0.58	2.73
5	5	2	DHT4-A	C7260	9	17.27	23.54	1.18	7.65	0.39	2.55
5	5	2	DHT4-A	D7255	10	19.13	26.57	1.87	5.05	0.59	1.60
5	5	2	Uncoated Alcamizer 1	B7750	9	18.52	6.44	4.12	2.76	1.37	0.92
5	5	2	Uncoated Alcamizer 1	C7260	10	20.82	9.25	2.51	3.57	0.80	1.13
5	5	2	Uncoated Alcamizer 1	D7255	10	17.13	20.15	3.42	5.32	1.08	1.68
5	5	3	Alcamizer 1	A7260	8	22.84	22.04	0.79	3.00	0.28	1.06
7.5	5	2	Alcamizer 1	B7750	20	19.73	19.62	1.53	10.45	0.34	2.34
7.5	5	2	Alcamizer 1	C7260	19	20.67	15.56	1.98	10.23	0.45	2.35
7.5	5	2	Alcamizer 1	D7255	20	17.83	24.20	1.58	5.17	0.35	1.16
7.5	5	2	DHT4-A	B7750	10	17.47	13.56	2.25	3.09	0.71	0.98
7.5	5	2	DHT4-A	C7260	10	19.56	15.00	1.34	3.23	0.42	1.02
7.5	5	2	DHT4-A	D7255	10	18.89	26.33	2.26	5.16	0.71	1.63
7.5	5	2	Uncoated Alcamizer 1	B7750	4	16.47	6.31	3.89	3.65	1.94	1.83
7.5	5	2	Uncoated Alcamizer 1	C7260	8	20.79	8.25	3.77	3.52	1.33	1.24
7.5	5	2	Uncoated Alcamizer 1	D7255	10	17.92	20.68	3.00	4.13	0.95	1.30
10	5	1	Alcamizer 1	A7260	7	18.74	17.83	0.64	3.16	0.24	1.20
10	5	1	DHT4-A	B7750	5	17.75	38.01	0.36	13.42	0.16	6.00
10	5	2	Alcamizer 1	A7260	9	21.34	19.01	0.44	1.88	0.15	0.63
10	5	2	DHT4-A	B7750	5	19.00	39.91	0.59	41.67	0.26	18.63
10	5	3	Alcamizer 1	A7260	10	21.42	19.83	0.75	3.37	0.24	1.07
15	5	2	DHT4-A	B7750	5	18.45	9.50	0.34	0.61	0.15	0.27
20	5	2	DHT4-A	B7750	5	18.00	8.24	0.24	0.85	0.11	0.38

neat case for all polymer grades and clay types, which indicates a poor interaction between the polymer and clay [2]. Depending on the polymer-clay system, either an increase or further decrease is observed at higher clay loadings (≥ 5 %wt). σ_{UTS} for HDPE B7750 (Fig. 2a) is less variable than for HDPE C7260 (Fig. 2b) and HDPE D7255 (Fig. 2c).

Considering the influence of the polymer grade, it is observed from the σ_{UTS} mean in Table II that neat HDPE B7750 has the highest mean with 22.26 MPa compared to HDPE C7260 (20.38 MPa) and HDPE D7260 (19.61 MPa). With the inclusion of clay it is observed that, in general, HDPE C7260 has a higher σ_{UTS} mean irrespective of clay type. For example, at a 2.5 wt% the mean σ_{UTS} is highest for HDPE C7260 (Alcamizer 1: 22.65 MPa, DHT4-A: 20.5

TABLE III: Percentage of Brittle, Intermediate and Ductile failures for the material system.

Failure	Neat	Alcamizer 1			DHT4-A					Uncoated Alcamizer 1			
		2.5 wt%	5 wt%	7.5 wt%	2.5 wt%	5 wt%	7.5 wt%	10 wt%	15 wt%	20 wt%	2.5 wt%	5 wt%	7.5 wt%
HDPE B7750													
Brittle	0	0	0	0	0	0	0	0	0	0	0	20	25
Intermediate	100	80	40	40	100	80	100	40	100	100	100	80	75
Ductile	0	20	60	60	0	20	0	60	0	0	0	0	0
HDPE C7260													
Brittle	0	0	0	50	0	0	0	0	0	0	20	0	0
Intermediate	100	60	25	25	60	100	100	0	0	0	80	100	100
Ductile	0	40	75	25	40	0	0	0	0	0	0	0	0
HDPE D725													
Brittle	0	0	0	0	0	0	0	0	0	0	20	0	0
Intermediate	100	80	40	60	60	100	60	0	0	0	80	100	100
Ductile	0	20	60	40	40	0	40	0	0	0	0	0	0

MPa and Uncoated Alcamizer 1: 20.17) compared to HDPE B7750 (Alcamizer 1: 19.96 MPa, DHT4-A: 18.71 MPa and Uncoated Alcamizer 1: 19.62) and HDPE D7255 (Alcamizer 1: 18.14 MPa, DHT4-A: 17.96 MPa and Uncoated Alcamizer 1: 18.16). Similar trends can be observed for the 5 and 7.5 wt%, with the exception of 5 wt% DHT4-A where HDPE B7750 has the highest mean (19.14 MPa) and HDPE C7260 the lowest (17.27 MPa).

Considering the influence of clay type within a polymer system, Alcamizer 1 generally provides a higher σ_{UTS} followed by DHT4-A and Uncoated Alcamizer. This is surprising, since Alcamizer 1 is known to be less compatible with HDPE than DHT4-A. The variability in the results for Uncoated Alcamizer is larger than Alcamizer and DHT4-A, with DHT4-A providing the least variability.

There is a lot of variation in the $\% \epsilon_f$ data as observed in Fig. 2d to 2f and as a result it is difficult to draw any sensible conclusions. The percentage failure based on the Brittle, Intermediate and Ductile grouping for the material system is summarised in Table III. Based on these groupings the majority of samples tend to fail in the Intermediate grouping, followed by Ductile and lastly Brittle. Potential material property degradation is observed with an increase in clay loading for HDPE C7260/Alcamizer 1 with 50% Brittle failures at 7.5wt% and HDPE B7750/Uncoated Alcamizer 1 with an increase in Brittle failures from 20% at 5 wt% to 25% at 7.5 wt%. This degradation in material properties is potentially due to increased agglomeration of particles [5].

Manufacturing and Testing System Influence

The influence of the manufacturing and testing system variables, discussed in Table I, on the mechanical properties of interest (i.e. σ_{UTS} and $\% \epsilon_f$) is shown in Fig. 3.

Effect of the Number of Extrusions

In Fig. 3a there is an initial increase in σ_{UTS} from 0 to 5 wt% followed by a decrease from 5 to 10 wt%. There is a clear increase in σ_{UTS} from 1 to 2 extrusions, whereas the change in σ_{UTS} from 2 to 3 extrusions is less significant. This observation is valid for all clay loadings. When considering the mean σ_{UTS} (c.f. Table II) for Neat HDPE A7260 there is an increase from 1 extrusion (18.80 MPa) to 2 extrusions (21.54 MPa) followed by a small increase to 3 extrusions (21.72 MPa). The increase in mean σ_{UTS} for 5 wt% Alcamizer is small with 22.28 MPa for 1 extrusion, 22.69 MPa for 2 extrusions and 22.84 MPa for 3 extrusions. Similar to the Neat HDPE A7260 the increase in σ_{UTS} for 10 wt% Alcamizer from 1 to 2 extrusions is 18.74 MPa to 21.34 MPa followed by a small increase to 21.42 MPa for 3 extrusions. There is a definite influence from 1 to 2 extrusions, where the mechanical property increases with an increase in the number of extrusions. However, when increasing the number of extrusions to 3 there is no advantage in the mechanical properties. Generally an increase in the number of extrusions causes a material to become more brittle which will influence the processability [5].

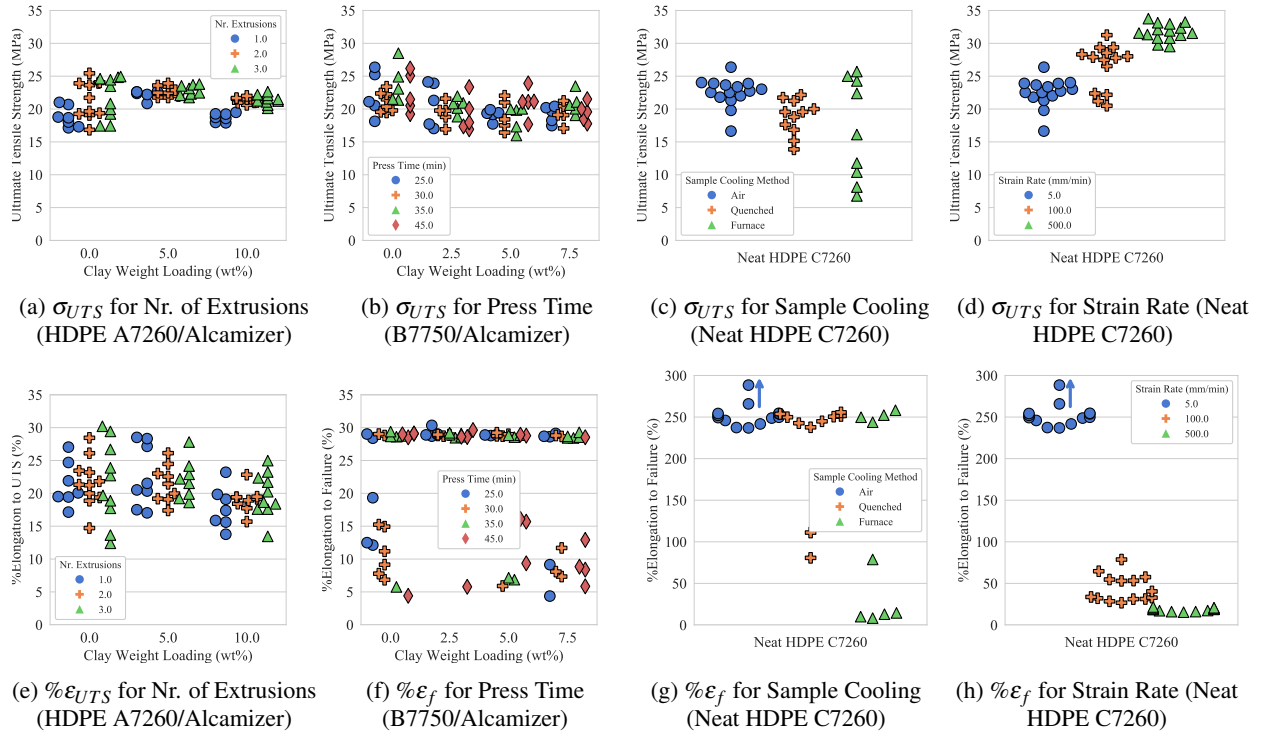


FIGURE 3: Influence of the Manufacturing and Testing System variables on σ_{UTS} and $\% \epsilon_f$.

There is more variability in the obtained results for the $\% \epsilon_{UTS}$, shown in Fig. 3e, than for σ_{UTS} . Not much can be said regarding the ductility of the material as the results were only available up to UTS.

Effect of Press Time

In Fig. 3b there is a decrease in σ_{UTS} as the clay weight loading increases, but σ_{UTS} appears to be constant from about 5 wt%. The change in press time has a larger influence on the neat samples where there is a decrease in the mean σ_{UTS} from 25 to 30 min (22.28 to 21.09 MPa), an increase to 35 min (23.87 MPa) followed by a decrease to 45 min (22.49 MPa). Once Alcamizer 1 is introduced at 2.5 wt% there is an increase in the mean σ_{UTS} with an increase in press time (25 min: 19.18 MPa, 30 min: 19.36 MPa, 35 min: 20.17 MPa) followed by a decrease to a mean σ_{UTS} of 19.12 MPa at 45 min. At 5 wt% the mean σ_{UTS} increases with an increase in press time (25 min: 18.45 MPa, 30 min: 19.08 MPa, 35 min: 19.28 MPa, 45 min: 20.98 MPa). Whereas at 7.5 wt% the mean σ_{UTS} initially increases from 25 min (18.10 MPa) to 30 min (19.38 MPa) followed by a decrease to 35 min (18.74 MPa) before increasing at 45 min (19.46 MPa). The increase in mean σ_{UTS} for an increase in press time is not considered enough of an improvement to warrant the additional time and effort required to press the tensile samples during manufacturing.

The $\% \epsilon_f$ in Fig. 3f does not provide additional insight into the influence of press time as there is no observable trend in the data. The percentage of Brittle, Intermediate and Ductile failures for an increase in press time are summarised in Table IV. Based on the failure criteria, most samples tend to fail within the Intermediate and Ductile regimes, with a few failing as Brittle. There appears to be no clear trend regarding the material failure with an increase in press time.

Effect of Sample Cooling Method

As noted in Fig. 3c the furnace cooled samples had the largest variance within the data set and also provided the lowest mean σ_{UTS} (16.73 MPa), whereas the air cooled samples provided the highest σ_{UTS} (22.51 MPa) and the quenched

TABLE IV: Percentage of Brittle, Intermediate and Ductile failures for Press Time.

Failure	Neat	2.5 wt%	5 wt%	7.5 wt%
25 min Press Time				
Brittle	0	20	0	0
Intermediate	100	80	40	40
Ductile	0	0	60	60
30 min Press Time				
Brittle	12.5	0	0	20
Intermediate	87.5	60	60	40
Ductile	0	40	40	40
35 min Press Time				
Brittle	0	0	0	0
Intermediate	60	100	40	0
Ductile	40	0	60	100
45 min Press Time				
Brittle	20	20	0	0
Intermediate	0	80	60	80
Ductile	80	0	40	20

TABLE V: Percentage of Brittle, Intermediate and Ductile failures for Sample Cooling Method.

Failure	Air	Quenched	Furnace
Brittle	0	0	11.11
Intermediate	0	0	33.33
Ductile	100	100	55.56

TABLE VI: Percentage of Brittle, Intermediate and Ductile failures for Strain Rate.

Failure	5 mm/min	100 mm/min	500 mm/min
Brittle	0	0	0
Intermediate	0	71.43	100
Ductile	100	28.57	0

sample mean σ_{UTS} was 19.33 MPa. The air cooled samples were also highly ductile with $\% \epsilon_f$ higher than 200% as seen in Fig. 3g, with the level of ductility decreasing for the quenched and furnace samples. This is to be expected as the method of cooling the tensile samples influences the material crystallinity which consequently influences its ductility. The percentage of Brittle, Intermediate and Ductile failure is provided in Table V, where it is clear that for the air cooled and quenched samples the primarily ductile, confirming the visual inspection of $\% \epsilon_f$. For the furnace samples the material ductility decreased with 11.11% Brittle failures, 33.33% Intermediate and 55.56% Ductile. This indicates that there is a degradation of the material properties of samples cooled using a furnace.

Effect of Strain Rate

In Fig. 3d a linear increase in σ_{UTS} is observed with an increase in strain rate where the mean σ_{UTS} is 20.38 MPa for 5 mm/min, 26.41 MPa for 100 mm/min and 31.73 MPa for 500 mm/min. The material ductility decreases as $\% \epsilon_f$ decreases with an increase in strain rate as shown in Fig. 3h. The percentage Brittle, Intermediate and Ductile failures are summarised in Table VI where there is a decrease in material ductility from 100% Ductile failures at 5 mm/min to 28.57% Ductile and 71.43% Intermediate failures at 100 mm/min and 100% Intermediate failures at 500 mm/min. The strain rate clearly has an influence on the mechanical properties. During tensile testing as the sample is pulled in tension the polymer chains extend. When considering a low strain rate the polymer chains have ample time to extend and therefore tend to exhibit more ductile behaviour. On the other hand at higher strain rates the polymer chains don't have enough time to extend and tend to break quickly resulting in a brittle behaviour.

STATISTICAL ANALYSIS

Considering that variability and uncertainty are expected in any manufacturing process, the aim is to quantify the sources of variability i.e., the percentage of variability attributable to the material system, the manufacturing methodology and random error.

From Table II it is observed that the number of observations per condition is fairly large, and the standard error of the mean is quite small compared to the mean for each response. This provides an indication that it should be possible to quantify statistically significant effects of the experimental variables, and significant differences between the levels of the variables of interest.

The results from the exploratory data analysis indicated a large variability in the observed responses for the $\% \epsilon_f$ response variable, which is also noted in Table II. As a result the $\% \epsilon_f$ was grouped into three categories as discussed

TABLE VII: Analysis of variance for σ_{UTS} . A significant effect is when $p < 0.05$.

	Df	Sum Sq	Mean Sq	F-value	Pr(>F)
Polymer.Grade	3	1084	361.4	53.419	<2e-16 ***
Clay.Type	3	581	193.6	28.617	<2e-16 ***
Clay.Loading	1	20	20.5	3.023	0.0826 .
Extrusions	2	47	23.7	3.505	0.0307 *
Strain.Rate	1	1539	1539.2	227.485	<2e-16 ***
Press.Time	1	4	4.5	0.663	0.416
Residuals	564	3816	6.8		

Significance Codes: '***': 0-0.001, '**': 0.001-0.01, '*': 0.01-0.05, '.' : 0.05-0.1, ' ' : 0.1-1.0

TABLE VIII: Linear model for σ_{UTS} (Type2: Alc1 = Alcamizer 1; DHT4 = DHT4-A; Neat = Neat; UAlc1 = Uncoated Alcamizer 1). A significant effect is when $p < 0.05$.

	Estimate	Std. Error	t-value	Pr(> t)
(Intercept)	20.254431	0.737995	27.445	< 2e-16 ***
Polymer.GradeB7750	-1.792339	0.55417	-3.234	0.00129 **
Polymer.GradeC7260	-0.921277	0.545691	-1.688	0.09191 .
Polymer.GradeD7255	-2.97388	0.572045	-5.199	2.81E-07 ***
Type2DHT4	-1.033413	0.328535	-3.146	0.00175 **
Type2Neat	0.021626	0.385145	0.056	0.95524
Type2UAlc1	-0.930066	0.352656	-2.637	0.00859 **
Clay.Loading	-0.092029	0.045094	-2.041	0.04174 *
Extrusions2	1.45562	0.628646	2.315	0.02094 *
Extrusions3	1.720001	0.708387	2.428	0.01549 *
Strain.Rate	0.022229	0.001478	15.043	< 2e-16 ***
Press.Time	0.015219	0.018697	0.814	0.41602

Significance Codes: '***': 0-0.001, '**': 0.001-0.01, '*': 0.01-0.05, '.' : 0.05-0.1, ' ' : 0.1-1.0

in the Data Processing section. The only case omitted is the data obtained during 2016 as the $\% \epsilon_f$ was only up to UTS as discussed in the Data Processing section and therefore not a meaningful measure. Based on this grouping there are 17 Brittle, 323 Intermediate and 157 Ductile samples.

The effects of polymer grade, clay type, clay loading, number of extrusions, press time and strain rate on the responses, σ_{UTS} and failure mode, were of interest. For the statistical analysis sample cooling was not considered as the data was confounded with only 9 furnace samples and 12 quenched samples compared to the majority of data using air cooled samples. Note due to the unbalanced nature of the experimental study, only the main or linear effects of the variables could be statistically quantified, but no two-order interactions.

Statistical Analysis for σ_{UTS}

The effects of the variables on the observed σ_{UTS} are quantified through the analysis of variance (ANOVA) [18]. The ANOVA results are given in Table VII for σ_{UTS} . The ANOVA quantifies the magnitude of the mean sum of squares of the variables compared to the mean sum of squares for random error, in other words it is a statistical test to determine whether two population means are equal. This ratio, referred to as the calculated F value, is compared to the F distribution with specified degrees of freedom for the variable of interest and the residual degrees of freedom. The p-value (Pr(>F)) in Table VII, is the probability that the effect of the variable on the response is only by chance. Therefore, the smaller the p-value the greater the probability that the variable has an effect on the response. For example, a p-value smaller than 0.05 indicates that the variable has a significant effect on the response with more than 95% confidence. From Table VII, it is clear that polymer grade, clay type, number of extrusions and strain rate have a statistically significant effect on σ_{UTS} with at least 97% confidence. Clay loading and press time don't have any significant effect on σ_{UTS} .

Table VIII provides the linear or regression model for σ_{UTS} as a function of the experimental variables. The $R^2 = 0.45$ is very low which indicates that the model cannot be used for predicting σ_{UTS} , whereas the standard error of the model is equal to 2.6, which is very small compared to the overall mean of 20.25 (first entry in Table VIII (Intercept)) and indicates that statistically significant effects of the variables on the σ_{UTS} can be quantified. The values in the Estimate column for the variables are the average change in σ_{UTS} for the specific level of the variable compared to its first level in alphabetical order. For example, σ_{UTS} is on average -1.79 units lower for polymer grade B7750 compared to grade A7260. For clay type, σ_{UTS} is on average 0.02 units higher for clay type Neat compared to type Alcamizer 1. Clay loading has on average a negative effect on σ_{UTS} i.e., σ_{UTS} decreases by 0.09 units on average for a one wt% increase in loading and σ_{UTS} increases by 0.022 units on average for a one mm/min increase in strain rate. From Table VIII, the p-value (Pr(> |t|)), indicates the probability that the effect is only by chance. All the effects of the variables are statistically significant with 95% confidence except for polymer grade C7260 which

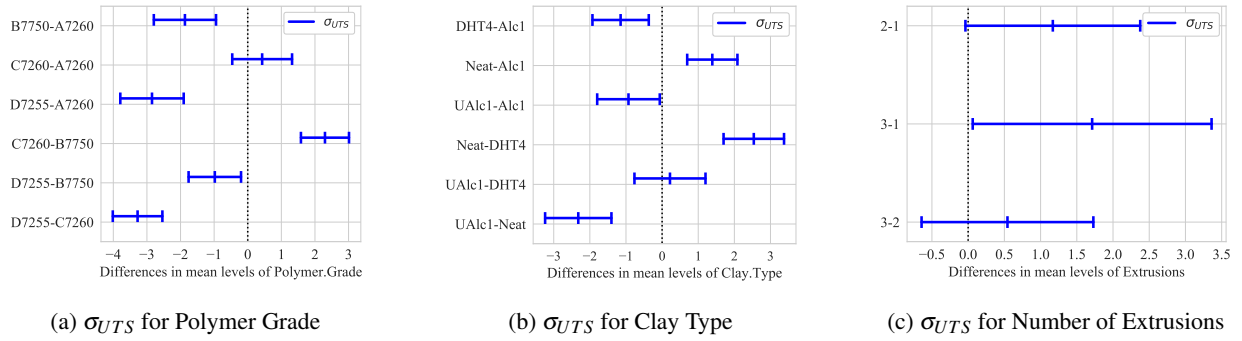


FIGURE 4: Tukey HSD test considering the statistical significance for different material and manufacturing variables on the σ_{UTS} .

is not statistically significantly different from A7260 at the 5 wt% level; the neat HDPE which is not statistically significantly different from Alcamizer 1; and press time which also has no statistically significant effect. Note for the linear model the statistical significance is quantified by comparing the ratio of the estimate to its standard error for the variable to a t-distribution with specified degrees of freedom [18].

The linear model in Table VIII does not provide a statistical comparison of all the variable levels with each other. The Tukey Honest Significant Difference (HSD) test can be performed to provide multiple comparisons between all the levels of the variable of interest [18]. The Tukey HSD is based on the Studentized Range Statistic and provides a family-wise true significant difference test. Fig. 4 provides the Tukey HSD tests between the levels for the different explanatory variables (polymer grade, clay type and number of extrusions). The intervals on the graph are 95% confidence intervals for the mean difference in σ_{UTS} between the two grades of interest. If the confidence interval does not contain zero, then the two levels of interest are statistically significantly different with 95% confidence. Note these are true 95% confidence intervals adjusted for the number of levels being compared.

From Fig. 4a, all the levels of polymer grade are statistically significantly different from each other, except grade C7260 which is not different from A7260 for the mean σ_{UTS} . In Fig. 4b all the clay types are statistically significantly different from each other, except for the Uncoated Alcamizer 1 and DHT4-A. From Fig. 4c there is only a statistically significant difference between 1 and 3 extrusions.

Statistical Analysis for Failure Mode

Due to the grouping of failure mode into Brittle, Intermediate and Ductile, the response variable has changed from a quantitative variable to a categorical variable, for which a different statistical technique is needed. A Linear Discriminant Analysis (LDA) technique is used which constructs linear functions of the variables which best separates the groups using a linear boundary [19]. The boundary is fixed by coefficients, denoted as LD1 and/or LD2 (depending on the linear model), and the value of these coefficients indicates the degree of influence of a design variable on the defined groupings.

The data for the number of extrusions were limited with 5 observations for 1 Extrusion, 492 for 2 Extrusions and 0 for 3 Extrusions. Similarly for the strain rate, there are 15 observations for 100 and 500 mm/min and 546 for 5 mm/min. Due to the skewed number of observations the number of extrusions and strain rate were therefore omitted from the analysis.

LDA considering all three groups

The failure mode group means for the three groups are shown in Table IX considering the different design variables. An LDA model is developed by modelling the failure mode grouping as a function of the polymer grade, clay type, clay loading and press time. Both the LD1 and LD2 coefficients from the LDA model are shown in Table X. The prior probabilities used in the analysis are 0.039 for Brittle, 0.661 for Intermediate and 0.299 for Ductile, which are the proportions of observations in the dataset.

TABLE IX: Means for the different groups of failure mode.

	Brittle	Intermediate	Ductile
Polymer.GradeC7260	0.4	0.331	0.548
Polymer.GradeD7255	0.2	0.331	0.209
Clay.TypeDHT4-A	0	0.280	0.139
Clay.TypeNeat	0.133	0.197	0.322
Clay.TypeUncoatedAlcamizer1	0.6	0.244	0
Clay.Loading	4.5	4.390	3.565
Press.Time	32.667	30.571	31.913

TABLE XI: Confusion matrix for the training data set with a 71.35% accuracy.

Predicted	Observed		
	Brittle	Ductile	Intermediate
Brittle	0	0	0
Ductile	3	39	19
Intermediate	12	76	235

TABLE X: Coefficients for the linear discrement model for failure mode.

	LD 1	LD 2
Polymer.GradeC7260	0.86269	-0.32056
Polymer.GradeD7255	0.01665	0.82452
Clay.TypeDHT4-A	-1.29087	1.55080
Clay.TypeNeat	-0.39885	0.09513
Clay.TypeUncoatedAlcamizer1	-2.68734	-1.04955
Clay.Loading	-0.00007	-0.04759
Press.Time	0.00264	-0.05430

TABLE XII: Confusion matrix for the testing data set with a 65.49% accuracy.

Predicted	Observed		
	Brittle	Ductile	Intermediate
Brittle	0	0	0
Ductile	0	9	4
Intermediate	2	33	65

It should be noted that HDPE B7750 and Alcamizer 1 are used as the baseline for the categorical variables polymer grade and clay type, respectively. Thus, they don't appear in Table X. The high values for the clay type coefficients (both LD1 and LD2) indicate that clay type has the largest influence on material failure groupings.

The LDA model is trained and tested to determine its ability to correctly predict the different groups. The data set is divided into a training set and a test set, with 80% of the data randomly selected for training the model and the remaining 20% used for testing. The confusion matrix for both the training and test set is shown in Tables XI and XII where the columns are the observed group and the rows the predicted group. For example, when considering the training set no Brittle groups were predicted, but 3 observations that should be Brittle were predicted as Ductile and 12 as Intermediate. Considering the testing set, Table XII, the model struggles to accurately predict the Brittle and Ductile classes and only achieved an accuracy of 71.35% for the training data set and 65.49% for the test data set. The poor performance of the model to accurately predict the Brittle and Ductile groups may be due to the few observations available for these groups compared to the Intermediate group.

LDA omitting the Brittle group

In the second LDA analysis, it was decided to omit the Brittle group as there were only 17 observations in the data set and based on the previous section the LDA does struggle to correctly predict an observation as Brittle. The resulting failure mode group means considering only Intermediate and Ductile is shown in Table XIII. The prior probabilities for the Ductile and Intermediate groups are determined based on their proportions of the observations in the dataset and are determined as 0.318 and 0.682, respectively.

Based on the resulting linear discriminant coefficient (LD1), again it is observed that clay type has the highest value which indicates that it has the largest influence on the material failure groupings. As in the previous analysis, the dataset was divided into a training and test data set by randomly selecting 80% of the data for training and using the remaining 20% for testing the model. The resulting confusion matrices are shown in Tables XV and XVI. In this case both the training and test data set resulted in a 72.92% accuracy and it is observed that the model is able to correctly group Intermediate where 52 observations were grouped correctly and only 17 were grouped as Ductile. Similarly, 18 observations were correctly grouped as Ductile and only 9 were incorrectly grouped as Intermediate for the test data set.

TABLE XIII: Means for the different groups of failure mode omitting the Brittle group.

	Intermediate	Ductile
Polymer.GradeC7260	0.324	0.574
Polymer.GradeD7255	0.317	0.189
Clay.TypeDHT4-A	0.298	0.115
Clay.TypeNeat	0.221	0.369
Clay.TypeUncoated Alcamizer 1	0.218	0
Clay.Loading	4.323	3.381
Press.Time	30.706	31.393

TABLE XV: Confusion matrix for the training data set with a 72.92% accuracy when omitting the Brittle group.

Predicted	Observed	
	Ductile	Intermediate
Ductile	63	45
Intermediate	59	217

TABLE XIV: Coefficients for the linear discrement model for failure mode omitting the Brittle group.

	LD 1
Polymer.GradeC7260	-0.935304341
Polymer.GradeD7255	0.13178971
Clay.TypeDHT4-A	1.757747774
Clay.TypeNeat	0.473677118
Clay.TypeUncoatedAlcamizer1	2.587720605
Clay.Loading	-0.018098249
Press.Time	0.005923164

TABLE XVI: Confusion matrix for the testing data set with a 72.92% accuracy when omitting the Brittle group.

Predicted	Observed	
	Ductile	Intermediate
Ductile	18	9
Intermediate	17	52

CONCLUSION

Over the past few years a number of experiments were performed which considered different polymer-clay composite systems and manufacturing parameters. This has largely been a one-factor-at-a-time experimental approach trying to understand the material system and manufacturing process and its influence on the mechanical properties of interest.

An exploratory data analysis (EDA) was considered to explore the historical data and to gain potential insight for the direction of future experimental work. The exploratory data analysis has shown that the material and testing system parameters have an influence on the mechanical properties of interest (σ_{UTS} and $\% \epsilon_f$). The results indicated that there is a poor interaction between the polymer and clay as σ_{UTS} tends to decrease with the addition of clay. A decrease in $\% \epsilon_f$ was also observed with an increase in clay loading. These observations correspond with what was expected based on literature [2, 4, 5]. The manufacturing parameters have slight influences but the small improvements in mechanical properties do not outweigh the substantial investment in time and resources required to achieve the improved properties. The strain rate had an observable influence on both the response variables.

To analyse and quantify the effect of these observations on both response variables, a statistical analysis was conducted. Initial observations in the statistical analysis indicated that no proper experimental designs were considered and as a result only main or linear effects between variables could be quantified. The statistical analysis for σ_{UTS} considered Analysis of Variance (ANOVA) and linear regression models. The ANOVA results indicated that polymer grade, clay type, number of extrusions and strain rate had a statistically significant effect on σ_{UTS} with at least 97% confidence. The linear regression model indicated polymer grade, clay type and clay loading had a negative effect on σ_{UTS} , while the number of extrusions and strain rate had a positive effect. To statistically analyse $\% \epsilon_f$ a Linear Discriminant Analysis (LDA) was considered. The results indicated that clay type has the largest influence on the material failure groupings.

Based on the observations of both the EDA and statistical analysis it is recommended that the following material, manufacturing and testing conditions are considered for future studies:

- **Polymer Grade:** The influence of polymer grade observed in the EDA is due to the different grades having different neat properties. The statistical analysis also indicated that there is no difference between the HDPE A7260 and HDPE C7260 grades. The EDA indicated that HDPE C7260 provides the highest σ_{UTS} .
- **Clay Type:** The EDA indicated that Alcamizer 1 resulted in higher σ_{UTS} compared to DHT4-A which was unexpected as Alcamizer 1 is not compatible with HDPE. There is also statistical significance for clay type and it should therefore be investigated further in future studies.
- **Clay Loading:** Clay loading should always be considered during a polymer clay study as it does have an effect on the mechanical properties. Based on the EDA and a previous study [9] it is suggested that clay loadings

considered not exceed 10 wt% as material degradation sets in.

- **Number of Extrusions:** There is statistical significance from 1 to 3 extrusions, but none from 2 to 3. This was also observed during the EDA and considering only 2 extrusions is therefore sufficient.
- **Press Time:** No statistical significance was found and the EDA indicated that the σ_{UTS} improvement from 25 to 45 min does not warrant the additional manufacturing time and cost. A press time of 25 min is therefore adequate.
- **Sample Cooling:** The EDA indicated that air cooled samples provide the highest mechanical properties and also no additional resources or cost during manufacturing.
- **Strain Rate:** Both analyses indicated a clear influence due to strain rate which should be further investigated.

In any future study, a better approach to designing the experimental studies is required to fully characterise, understand and quantify the effects on the mechanical properties of the polymer-clay composite. The conclusions and recommendations from this study can be used to determine the parameters and ranges of interest for future experimental designs.

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