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Making road base and foundation from secondary waste minerals and recycled aggregates

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Making Road Base and Foundation from Secondary Waste Minerals and Recycled Aggregates

By

Kande Bure Bai Kamara

January 2020



A thesis submitted in partial fulfilment of the University's requirements for the Degree of Doctor of Philosophy in Civil Engineering, Architecture and Building Content removed on data protection grounds



Certificate of Ethical Approval

Applicant:

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Project Title:

Making Road Base and Foundation from Secondary Waste Minerals and Recycled Aggregates

This is to certify that the above named applicant has completed the Coventry University Ethical Approval process and their project has been confirmed and approved as Low Risk

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CHAPTER	CONTENTS				
	List o	of Figures		xiii	
	List o	of Tables		xvii	
	Acro	nyms and	Abbreviations	xix	
	List	of Confere	nces and Papers Published	xxiii	
	ABS	RACT		xxiv	
	ACK	NOWLEDG	GEMENTS	xxvi	
1.0	INTR	ористіоі	N	1	
	1.1	Introduct	ion	1	
	1.2	Backgrou	und Information	1	
		1.2.1 T 1.2.2 U 1.2.3 S	he World on Climate Change JK on Climate Change Sustainable Development	2 3 4	
	1.3	Statistics and Seco	Statistics on Construction Waste, Recycled and Secondary Waste Aggregates		
		1.3.1 S 1.3.2 S W	statistics on Construction Waste statistics on Recycled and Secondary Vaste Aggregates	5 5	
	1.4	Challeng Aggregat	les and Benefits of the use of Secondary tes and Recycled Aggregates	6	
		1.4.1 W 1.4.2 W 1.4.3 C	Vhat are Secondary Aggregates Vhat are Recycled Aggregates Challenges to the use of Recycled	6 7	
		a 1.4.4 B S	nd Secondary Waste Minerals Senefits to the use of Recycled and Secondary Waste Minerals	7 8	
	1.5	Aim and	Objectives of Study	9	
		1.5.1 A 1.5.2 O 1.5.3 T 1.5.4 R	im Objectives The Research Questions Research Gap and Novelty	9 9 10 10	
2.0	MET	HODOLOG	βY	12	
	2.1	Methodo	logy Overview	12	
		2.1.1 R	Report Structure	13	

	2.2	Initial	Research in Identifying Materials for Study	14
		2.2.1 2.2.2	Initial Primary Research Initial Secondary Research	14 14
	2.3	Identif	ying Materials for the Research Study	15
		2.3.1 2.3.2 2.3.3 2.3.4 2.3.5	Aggregates and Quarry Industry Cement Factory Chemical Industry Steel Industry Minerals and Aggregate Recycling Centres	17 19 19 20 21
			2.3.5.1 Day Aggregates 2.3.5.2 KSD Recycled Aggregates Limited	21 21
			2.3.5.3 Raymond Brown Minerals and Recycling Limited 2.3.5.4 Plasterboard Company	22 22
		2.3.6	Analysis of Data from Initial Secondary Research	22
3.0	LITER	ATURE	REVIEW	25
	3.1	Ceme	nt Replacement Materials in Concrete Pavement	25
	3.2	Potent	ial use of By-product in Road Construction	26
	3.3	Previo Concr	us use of By-product in Hydraulically Bound ete Pavement	27
		3.3.1	By-product use in Pavement in the United	27
		3.3.2	By-product use in Pavement in the European	30
		3.3.3	By-product use in Pavement in the USA	31
	3.4	Enviro Paven	nmental Effects in using By-product in Concrete nent	34
		3.4.1	Environmental Impact in using By-product	34
	3.5	Base I	Materials Available in the Market	34
		3.5.1 3.5.2 3.5.3	By-pass Dust Steel Slag Dust Gypsum	34 35 36
			3.5.3.1 Plasterboard Gypsum 3.5.3.2 Flue Gas Desulphurisation 3.5.3.3 Red Gypsum 3.5.3.4 Phosphogypsum	36 41 43 45
		3.5.4	Reclaimed Asphalt Fillers / Aggregates	45
		3.5.5	Quarry Waste Dust	47

		3.5.6	Limes	tone		48
			3.5.6.1 3.5.6.2 3.5.6.3	Production of Uses of Lime Limestone Ch	Limestone in Great Britain stone nemical Formula – Thermal	49 49
				Decompositio	n and Hydration	50
				3.5.6.3.1	Thermal Decomposition of Limestone	50 51
				3.5.6.3.3	Chemical Composition of Dolomite	52
			3.5.6.4	4 Lime Kiln Dus	and Hydrated Lime	52
		3.5.7	Groun	d Granulated E	Blast Furnace Slag	55
	3.6	Summ	nary of F	Review		55
4.0	LABO	RATOF	RY EXP		N MATERIALS SUITABILITY	57
	4.1	Introdu	uction			57
	4.2	Testing Methodology for By-product Suitability				
	4.3	Chem	ical Cor	mposition of Ma	aterials	58
		4.3.1 4.3.2 4.3.3 4.3.4	Chemi Chemi Chemi Chemi	ical Properties ical Properties ical properties ical Properties	of By-pass Dust of Steel Slag Dust of Plasterboard Gypsum of Ground Granulated Blast	58 59 59
		4.3.5	Furna Chemi Hydrat	ce Slag ical Properties ted Tailing	of Lime Kiln Dust and Large	60 60
	4.4	Chem	ical Cor	nposition of Ot	her Materials used in this Research	61
		4.4.1	Chemi	ical Properties	of BPD-C	61
			4.4.1.1	1 X-Ray Fluore	scence Analysis of BPD-C	61
		4.4.2	Chemi	ical Properties	of SSD	62
			4.4.2.2 4.4.2.2	1 X-Ray Diffrac 2 X-Ray Fluore	tion Analysis of SSD scence Analysis of SSD	62 63
		4.4.3	Chemi	ical Properties	of PG	63
			4.4.3.2 4.4.3.2	1 X-Ray Diffrac 2 X-Ray Fluore	tion Analysis of PG scence Analysis of PG	63 64
		4.4.4	Chemi	ical Properties	of V-B5G	64
			4.4.4.1	1 X-Ray Diffrac	tion Analysis of V-B5G	64

65
65 66
66
66 67
67
67 68
68
S 68
sign 68
of 68
s 69
70
70 the 71 72 73 74 75 76 77
70 the 71 72 73 74 75 76 77 79
70 the 70 71 72 73 74 75 76 77 79 79
70 the 71 72 73 74 75 76 77 79 79 79
70 the 70 71 72 73 74 75 76 77 79 79 79 6ers 79 b 79
the 70 71 72 73 74 75 76 77 79 79 79 6ers 79 b 79 80

5.0

		 5.3.3 Steel Slag Dust (SSD) Preparation 5.3.4 Plasterboard Gypsum (PG) Preparation 5.3.5 Vitamin B5 Gypsum (V-B5G) Preparation 5.3.6 Reclaimed Asphalt Filler (RAF) Preparation 5.3.7 Quarry Waste Dust (QWD) Preparation 5.3.9 Ground Granulated Blast Furnace Preparation 	on 81 aration 82 paration 82 reparation 83 aration 83 Preparation 83 e Slag (GGBS) 83
	5.4	Mixing of Binder Samples	83
	5.5	Weighing and Compaction of Samples	84
	5.6	Casting and Curing of Test Specimens	86
	5.7	Testing of Specimens	87
6.0	PHA: RESI	E ONE AND PHASE TWO EXPERIMEN	TAL TESTS AND 89
	6.1	Introduction	89
	6.2	Mix Designs of Cement Binders	89
		6.2.1 Group 1 – PG/MHT/GGBS 6.2.2 Group 2 – PG/RAF/GGBS 6.2.3 Group 2A – V-B5G/RAF/GGBS 6.2.4 Group 3 – PG/GGBS/BPD-C 6.2.5 Group 4 – PG/GGBS/BPD-H 6.2.6 Group 5 – V-B5G/MHT/SSD 6.2.7 Group 6 – V-B5G/RAF/SSD 6.2.8 Group 7 – V-B5G/SSD/BPD-C 6.2.9 Group 8 – V-B5G/SSD/BPD-H 6.2.10 Group 9 – PG/QWD/GGBS 6.2.11 Group 9A – V-B5G/QWD/GGBS	89 90 91 92 93 94 95 96 97 98 99
	6.3	Compressive Strength Results	100
		6.3.1 Group 1 Mixture Design Results	100
		 6.3.1.1 Mix Proportions and Com Strengths 6.3.1.2 Contour Plots for 7 and 28 Compressive Strength 6.3.1.3 Comparisons of 7 and 28 Compressive Strength 	pressive 100 3 Days 101 Days 102
		6.3.2 Group 2 Mixture Design Results	102
		 6.3.2.1 Mix Proportions and Com Strengths 6.3.2.2 Contour Plots for 7 and 28 Compressive Strength 6.3.2.3 Comparisons of 7 and 28 Compressive Strength 	pressive 102 3 Days 103 Days 104
		-	

6.3.3	Group 2A Mixture Design Results	105
	 6.3.3.1 Mix Proportions and Compressive Strengths 6.3.3.2 Contour Plots for 7 and 28 Days Compressive Strength 6.3.3.3 Comparisons of 7 and 28 Days 	105 105
621	Croup 2 Mixture Design Bosults	100
0.3.4		107
	 6.3.4.1 Mix Proportions and Compressive Strengths 6.3.4.2 Contour Plots for 7 and 28 Days Compressive Strength 	107 108
	6.3.4.3 Comparisons of 7 and 28 Days Compressive Strength	109
6.3.5	Group 4 Mixture Design Results	110
	6.3.5.1 Mix Proportions and Compressive Strengths	110
	Compressive Strength	110
	Compressive Strength	111
6.3.6	Group 5 Mixture Design Results	112
	 6.3.6.1 Mix Proportions and Compressive Strengths 6.3.6.2 Contour Plots for 7 and 28 Days Compressive Strength 6.3.6.3 Comparisons of 7 and 28 Days Compressive Strength 	112 113 114
6.3.7	Group 6 Mixture Design Results	114
	 6.3.7.1 Mix Proportions and Compressive Strengths 6.3.7.2 Contour Plots for 7, 28 and 90 Days Compressive Strength 6.3.7.3 Comparisons of 7, 28 and 90 Days Compressive Strength 	114 115 116
6.3.8	Group 7 Mixture Design Results	117
	 6.3.8.1 Mix Proportions and Compressive Strengths 6.3.8.2 Contour Plots for 28, 90 and 180 Days Compressive Strength 6.3.8.3 Comparisons of 28, 90 and 180 Days 	117 118
	Compressive Strength	119

	6.3.9	Group 8 Mixture Design Results	120
		 6.3.9.1 Mix Proportions and Compressive Strengths 6.3.9.2 Contour Plots for 7, 28 and 90 Days Compressive Strength 6.3.9.3 Comparisons of 7, 28 and 90 Days Compressive Strength 	120 120 122
	6.3.10	Group 9 Mixture Design Results	123
		 6.3.10.1 Mix Proportions and Compressive Strengths 6.3.10.2 Contour Plots for 28, 90 and 180 Days Compressive Strength 6.3.10.3 Comparisons of 28, 90 and 180 Days Compressive Strength 	123 123 125
	6.3.11	Group 9A Mixture Design Results	126
		 6.3.11.1 Mix Proportions and Compressive Strengths 6.3.11.2 Contour Plots for 28 and 90 Days Compressive Strength 6.3.11.3 Comparisons of 28 and 90 Days Compressive Strength 	126 126 128
	6.3.12	Binary Group – PG/GGBS	128
6.4	Phase	Two - Validation of Prediction for Chosen Groups	130
	6.4.1 6.4.2 6.4.3 6.4.4	Understanding Coefficient Equation Group 2 Validation of Prediction Group 2A Validation of Prediction Group 9 Validation of Prediction	130 130 132 133
6.5	Phase	Two - Refining the Chosen Groups	135
PHAS		AND TWO ANALYSIS AND DISCUSSIONS	137
7.1	Introdu	iction	137
7.2	Evalua The Cł	tion of Hydration and Pozzolanic Effects on nosen Groups	137
7.3	The Ef Mediur	fect of Different Liquid / Solid Ratios on Short to n-term Compressive Strength	139
7.4	The Ef Long-te	fect of Different Liquid / Solid Ratios on erm Compressive Strength	141
7.5	The Ef and G(fect of PG and V-B5G in Mixes Containing RAF GBS	143
7.6	The Ef	fect of RAF and QWD in Mixes Containing PG	

7.0

		and G	GBS	145	
	7.7	Streng	th Development of Hydraulic Paste	146	
	7.8	Discus	sions	148	
8.0	PHAS THE C	e thre Ptimu	EE EXPERIMENTAL TESTS AND CHOOSING M BLEND	151	
	8.1	Introdu	uction	151	
	8.2	Frost S	Susceptibility – Resistance to Freezing and Thawing	151	
	8.3	High P	Pressure Permeability Test on Preferred Mixes	152	
	8.4	Resista	ance to Freezing and Thawing Results	154	
	8.5	High P	Pressure Flow Test Results	155	
	8.6	Choos	ing the Optimum Blend	156	
9.0	PHAS CONC	E FOUF RETE	R EXPERIMENTAL TESTS ON SUSTAINABLE	159	
	9.1	Introdu	uction	159	
	9.2	Sustainable Concrete			
		9.2.1	WB Concrete with Natural Fines and Coarse Aggregates	159	
			9.2.1.1 Mix Proportions for Experiments on WB Concrete With Natural Fine and Coarse Aggregate	160	
		9.2.2	WB Concrete with Cement as Supplementary Material	160	
			9.2.2.1 Reviews on Supplementary Mixes9.2.2.2 Mix Proportions for Experiments on Supplementary Mixes	160 162	
		9.2.3	WB Concrete with Crushed Recycled Aggregates	162	
			 9.2.3.1 Reviews on Recycled Concrete Aggregates 9.2.3.2 Mix Proportions for Experiments on WB Concrete with Recycled Concrete as Fines / Coarse Aggregates and Combination of Recycled Concrete 	162	
			and Natural Aggregates	163	
			Preparation of Samples	164	
	9.3	Experi	mental Results of Sustainable Concrete	165	
		9.3.1	Results of WB Concrete with Natural Fines and Coarse Aggregates Results on WB Concrete with Comont as	165	
		9.J.Z	Supplementary Material	167	

		9.3.3	Results on WB Concrete with Recycled Concrete as Fines / Coarse Aggregates and Combination of Recycled Concrete and Natural Aggregates	168
			9.3.3.1 Compressive Strength 9.3.3.2 Water Absorption	169 170
	9.4	Worka	bility	173
	9.5	Analys	is of Sustainable Concrete	173
10.0	CONC	LUSIOI	NS	176
	10.1	Resea	rch Questions	176
	10.2	Resea	rch Objectives	179
		10.2.1 10.2.2 10.2.3 10.2.4	Objective 1 Objective 2 Objective 3 Objective 4	180 180 180 181
	10.3	Conclu	isions Summary	182
11.0	SUSTA AND R	AINABI ECOM	LITY, APPLICATIONS OF RESEARCH MATERIALS MENDATIONS FOR FURTHER STUDIES	184
	11.1	Introdu	iction	184
	11.2	Recom	mendations for Future Studies	184
	11.3	Applica	ations of Research Materials	185
REFERENCE	S			186
APPENDIX A	Questi	ionnair	e	200
APPENDIX B	Сору	of Corre	espondence via Companies Portal	201
APPENDIX C	Сору	of Emai	Is Response from Companies	205
APPENDIX D	Сору	of Emai	ils from Author in Response to Companies Emails	209

LIST OF FIGURES

Figure 2.1	Outline Methodology	12
Figure 2.2	Tarmac Mancetter Quarry – Atherstone, Warwickshire 2017	17
Figure 2.3	Tarmac Tunstead – Old Moor Quarry Complex 2017	18
Figure 2.4	Cemex Rugby Cement Plant – Rugby, Warwickshire 2012	19
Figure 2.5	Tarmac Port Talbot Quarry – Wales 2014	20
Figure 2.6	Binder Materials Obtained by Sectors	23
Figure 2.7	Binder Materials Obtained by Companies	24
Figure 3.1	Compositional Relationships between Cement and Cement	
	Replacement Materials	26
Figure 3.2	Coventry Hospital Plasterboard Material Flow Diagram	38
Figure 3.3	Chemical Reaction of Calcium Carbonate when Heated	51
Figure 3.4	Chemical Reaction of Quicklime and Water	51
Figure 3.5	Chemical Reaction of Calcium Carbonate and Magnesium	
	Carbonate	52
Figure 3.6	Lime Hydrating Plant	53
Figure 4.1	Methodology for Identifying Suitable By-product	58
Figure 4.2	XRD for Steel Slag Dust	62
Figure 4.3	XRD for Plasterboard Gypsum	63
Figure 4.4	XRD for Vitamin B-5 Gypsum	64
Figure 4.5	XRD for Reclaimed Asphalt Filler	65
Figure 4.6	XRD for Quarry Waste Dust	66
Figure 4.7	XRD for Medium Hydrating Tailing	67
Figure 4.8	Saturated Samples Ready to be Tested and Hanna Instrument	70
Figure 4.9	Particle Size Distribution of Raw Materials used in the Research	71
Figure 4.10	Particle Size Distribution of SSD using Malvern Mastersizer	72
Figure 4.11	Particle Size Distribution of PG using Malvern Mastersizer	73
Figure 4.12	Particle Size Distribution of V-B5G using Malvern Mastersizer	74
Figure 4.13	Particle Size Distribution of RAF using Malvern Mastersizer	75
Figure 4.14	Particle Size Distribution of QWD using Malvern Mastersizer	76
Figure 4.15	Particle Size Distribution of MHT using Malvern Mastersizer	77
Figure 4.16	Particle Size Distribution of GGBS using Malvern Mastersizer	78
Figure 5.1	Ball Milling machine	81
Figure 5.2	Plasterboard Broken Down into Sizeable Pieces	82
Figure 5.3	Sieving of plasterboard to remove paper	82
Figure 5.4	KENWOOD Machine with Sample in the Bowl ready to be Mixed	84

Figure 5.5	Weighing Scale and Plastic Containers used to Place 90g Sample	85
Figure 5.6	Gang of Three 50 mm Cube Moulds being Prepared for Use	85
Figure 5.7	Gang of Three 50 mm Mould Compacted to 90 KN	85
Figure 5.8	Avery Denison 500 Machine Linked to a Computer	85
Figure 5.9	Specimens being Cured in Propagators	86
Figure 5.10	Label showing Production Dates Mixes were Prepared	86
Figure 5.11	Lloyd LS – 50 Equipment Linked to Computer	87
Figure 5.12	Avery Denison 2000 Machine Linked to a Computer	88
Figure 6.1	Group 1 – Simplex Design Plot (PG/MHT/GGBS)	90
Figure 6.2	Group 2 – Simplex Design Plot (PG/RAF/GGBS)	91
Figure 6.3	Group 2A – Simplex Design Plot (V-B5G/RAF/GGBS)	92
Figure 6.4	Group 3 – Simplex Design Plot (PG/GGBS/BPD-C)	93
Figure 6.5	Group 4 – Simplex Design Plot (PG/GGBS/BPD-H)	94
Figure 6.6	Group 5 – Simplex Design Plot (V-B5G/MHT/SSD)	95
Figure 6.7	Group 6 – Simplex Design Plot (V-B5G/RAF/SSD)	96
Figure 6.8	Group 7 – Simplex Design Plot (V-B5G/SSD/BPD-C)	97
Figure 6.9	Group 8 – Simplex Design Plot (V-B5G/SSD/BPD-H)	98
Figure 6.10	Group 9 – Simplex Design Plot (PG/QWD/GGBS)	99
Figure 6.11	Group 9A – Simplex Design Plot (V-B5G/QWD/GGBS)	100
Figure 6.12	Group 1 – 7 Days Compressive Strength Contour Plot	101
Figure 6.13	Group 1 – 28 Days Compressive Strength Contour Plot	101
Figure 6.14	Group 1 – Comparisons of 7 and 28 Days Compressive	
	Strength	102
Figure 6.15	Group 2 – 7 Days Compressive Strength Contour Plot	103
Figure 6.16	Group 2 – 28 Days Compressive Strength Contour Plot	104
Figure 6.17	Group 2 – Comparisons of 7 and 28 Days Compressive	
	Strength	104
Figure 6.18	Group 2A – 7 Days Compressive Strength Contour Plot	106
Figure 6.19	Group 2A – 28 Days Compressive Strength Contour Plot	106
Figure 6.20	Group 2A – Comparisons of 7 and 28 Days Compressive	
	Strength	107
Figure 6.21	Group 3 – 7 Days Compressive Strength Contour Plot	108
Figure 6.22	Group 3 – 28 Days Compressive Strength Contour Plot	109
Figure 6.23	Group 3 – Comparisons of 7 and 28 Days Compressive	
	Strength	109
Figure 6.24	Group 4 – 7 Days Compressive Strength Contour Plot	111
Figure 6.25	Group 4 – 28 Days Compressive Strength Contour Plot	111

Figure 6.26	Group 4 – Comparisons of 7 and 28 Days Compressive	
	Strength	112
Figure 6.27	Group 5 – 7 Days Compressive Strength Contour Plot	113
Figure 6.28	Group 5 – 28 Days Compressive Strength Contour Plot	113
Figure 6.29	Group 5 – Comparisons of 7 and 28 Days Compressive	
	Strength	114
Figure 6.30	Group 6 – 7 Days Compressive Strength Contour Plot	115
Figure 6.31	Group 6 – 28 Days Compressive Strength Contour Plot	116
Figure 6.32	Group 6 – 90 Days Compressive Strength Contour Plot	116
Figure 6.33	Group 6 – Comparisons of 7, 28 and 90 Days Compressive	
	Strength	117
Figure 6.34	Group 7 – 28 Days Compressive Strength Contour Plot	118
Figure 6.35	Group 7 – 90 Days Compressive Strength Contour Plot	118
Figure 6.36	Group 7 – 180 Days Compressive Strength Contour Plot	119
Figure 6.37	Group 7 – Comparisons of 28, 90 and 180 Days Compressive	
	Strength	119
Figure 6.38	Group 8 – 7 Days Compressive Strength Contour Plot	121
Figure 6.39	Group 8 – 28 Days Compressive Strength Contour Plot	121
Figure 6.40	Group 8 – 90 Days Compressive Strength Contour Plot	122
Figure 6.41	Group 8 – Comparisons of 7, 28 and 90 Days Compressive	
	Strength	122
Figure 6.42	Group 9 – 28 Days Compressive Strength Contour Plot	124
Figure 6.43	Group 9 – 90 Days Compressive Strength Contour Plot	124
Figure 6.44	Group 9 – 180 Days Compressive Strength Contour Plot	125
Figure 6.45	Group 9 – Comparisons of 28, 90 and 180 Days Compressive	
	Strength	125
Figure 6.46	Group 9A – 28 Days Compressive Strength Contour Plot	127
Figure 6.47	Group 9A – 90 Days Compressive Strength Contour Plot	127
Figure 6.48	Group 9A – Comparisons of 28 and 90 Days Compressive Strength	128
Figure 6.49	Binary Group – Comparisons of 7 and 28 Days Compressive Strength	129
Figure 6.50	Group 2 – Predicted and Actual Compressive Strength at 28 Days	131
Figure 6.51	Group 2A – Predicted and Actual Compressive Strength at 28 Days	133
Figure 6.52	Group 9 – Predicted and Actual Compressive Strength at 28 Days	134
Figure 6.53	Group 2, 2A and 9 – Comparisons of Liquid/Solid Ratios at 28 Days	
	Strength	136
Figure 7.1	Group 2 – Comparison of Replacement of 13, 15 and 17% Liquid/Solid	
	Ratio	139

Figure 7.2	Comparisons of 13, 15 and 17% Liquid/Solid Ratio at 28 Days	
	Compressive Strength	140
Figure 7.3	Development of Compressive Strength on Selected Mixes Using	
	Liquid / Solid Ratio of 13, 15 and 17% at 7, 14, 28 and 90 days	143
Figure 7.4	Comparisons on the Effects of PG and V-B5G on Selected Mixes	
	Containing RAF and GGBS	144
Figure 7.5	Comparisons on the Effects of RAF and QWD on Selected Mixes	
	Containing PG and GGBS	146
Figure 7.6	Development of Compressive Strength as Specimens Age with	
	Liquid / Solid Ratio of 17%	147
Figure 7.7	Percentage of Strength Development	148
Figure 8.1	High Pressure Flow Test Layout	152
Figure 8.2	High Pressure Permeability of Preferred Mixes	156
Figure 8.3	Compressive Strength at 9, 28, and 90 Days of Top Three Mixes	157
Figure 8.4	Percentage of Strength Development of Mix 1 (Group 2), Mix 13	
	(Group 2A) and Mix 13 (Group 9)	158
Figure 9.1	Preparation of Equipment for Crushing	165
Figure 9.2	Crushed Samples	165
Figure 9.3	Comparison of WB Concrete with Control Mix	166
Figure 9.4	Comparison of Supplementary Mixes at 0.42 Ratio	167
Figure 9.5	Comparison of Supplementary Mixes at 0.50 Ratio	168
Figure 9.6	Comparison of RAC and BAC Mixes	170
Figure 9.7	Absorption Rate of WB with Recycled Concrete as Fines and	
	Coarse Aggregates	171
Figure 9.8	Absorption Rate of WB with Combination of Recycled Concrete and	
	Natural Fine and Coarse Aggregates	172
Figure 9.9	Particle Size Distribution of PG, QWD and GGBS	175

LIST OF TABLES

Table 2.1	Summary of Sectors Replied and Materials Supplied	16
Table 3.1	Summary of Potential Uses of By-product Materials in Concrete	
	Pavement	25
Table 3.2	Typical Properties of PFA, GGBS and Microsilica	26
Table 3.3	Provisions for the Use of Secondary and Recycled Materials	28
Table 3.4	Summary of Barriers and Mechanisms for Increasing Beneficial	
	Use of RMCs	32
Table 4.1	Chemical Oxides Composition (BPD)	59
Table 4.2	Chemical Oxides Composition (SSD)	59
Table 4.3	Chemical Oxides Composition (PG)	60
Table 4.4	Chemical Oxides Composition (GGBS)	60
Table 4.5	Chemical Oxides Composition of LKD and LHT	60
Table 4.6	XRF of BPD-C	61
Table 4.7	XRF of SSD	63
Table 4.8	XRF of PG	64
Table 4.9	XRF of V-B5G	65
Table 4.10	XRF of RAF	66
Table 4.11	XRF of QWD	67
Table 4.12	XRF of MHT	68
Table 4.13	XRF of GGBS	68
Table 4.14	pH Values of Materials used in the Phase 1 Experiments	70
Table 6.1	Group 1 – Mix Design (PG/MHT/GGBS)	89
Table 6.2	Group 2 – Mix Design (PG/RAF/GGBS)	90
Table 6.3	Group 2A – Mix Design (V-B5G/RAF/GGBS)	91
Table 6.4	Group 3 – Mix Design (PG/GGBS/BPD-C)	92
Table 6.5	Group 4 – Mix Design (PG/GGBS/BPD-H)	93
Table 6.6	Group 5 – Mix Design (V-B5G/MHT/SSD)	94
Table 6.7	Group 6 – Mix Design (V-B5G/RAF/SSD)	95
Table 6.8	Group 7 – Mix Design (V-B5G/SSD/BPD-C)	96
Table 6.9	Group 8 – Mix Design (V-B5G/SSD/BPD-H)	97
Table 6.10	Group 9 – Mix Design (PG/QWD/GGBS)	98
Table 6.11	Group 9A – Mix Design (V-B5G/QWD/GGBS)	99
Table 6.12	Group 1 – Compressive Strength (PG/MHT/GGBS)	100
Table 6.13	Group 2 – Compressive Strength (PG/RAF/GGBS)	103
Table 6.14	Group 2A – Compressive Strength (V-B5G/RAF/GGBS)	105

Table 6.15	Group 3 – Compressive Strength (PG/GGBS/BPD-C)	108
Table 6.16	Group 4 – Compressive Strength (PG/GGBS/BPD-H)	110
Table 6.17	Group 5 – Compressive Strength (V-B5G/MHT/SSD)	112
Table 6.18	Group 6 – Compressive Strength (V-B5G/RAF/SSD)	115
Table 6.19	Group 7 – Compressive Strength (V-B5G/SSD/BPD-C)	117
Table 6.20	Group 8 – Compressive Strength (V-B5G/SSD/BPD-H)	120
Table 6.21	Group 9 – Compressive Strength (PG/QWD/GGBS)	123
Table 6.22	Group 9A – Compressive Strength (V-B5G/QWD/GGBS)	126
Table 6.23	Binary Group – Compressive Strength (PG/GGBS)	129
Table 6.24	Group 2 – Predicted and Actual Strength	131
Table 6.25	Group 2A – Predicted and Actual Strength	132
Table 6.26	Group 9 – Predicted and Actual Strength	134
Table 6.27	Group 2 – Comparisons of 13, 15 and 17% liquid / solid ratio	
	compressive strength results at 28 days	136
Table 8.1	Resistance to freezing and thawing on Mixes 1 (Group 2), Mix 13	
	(Group 2A) and Mix 13 (Group 9)	155
Table 8.2	Strength Development of Selected Mixes on Groups 2, 2A and 9	158
Table 9.1	Mix Proportions of Concrete Mixtures – WB with Natural Fines and	
	Coarse Aggregates	160
Table 9.2	Mix Proportions of Concrete Mixtures – WB with Supplementary Mixes	162
Table 9.3	Mix Proportions of Materials	164
Table 9.4	Compressive Strength of WB and Control Mix at 7, 28 and 90 Days	166
Table 9.5	Compressive Strength of Supplementary Mixes at 7, 28 and 90 Days	167
Table 9.6	Compressive Strength of RAC / BAC and Control Mix at 7, 28 and	
	90 Days	169
Table 9.7	Chemical Oxides of Materials (PC Obtained from Neville 2012)	174

ACRONYMS AND ABBREVIATIONS

AASHTO	American Association of State Highway and Transportation Officials
ADS	Advanced Directional Sign
AMS	American Meteorological Society
ASTM	American Society for Testing Materials
BA	Bottom Ash
BAC	Blended Aggregate Concrete
BAC – WB	Blended Aggregate Concrete with Warwickshire Blend
BCS	Blend Calcium Sulphate
BFS	Blast Furnace Slag
BFSA	Blast Furnace Slag Aggregate
BGS	British Geological Survey
BOF	Basic Oxygen Furnace
BOS	Basic Oxygen Slag
BPD	By-pass Dust
BPD-C	By-pass Dust from Cemex
BPD-H	By-pass Dust from Hanson
CBGM	Cement Bound Granular Mixture
СВМ	Cement Bound Mixture
CBR	California Bearing Ratio
CCBs	Coal Combustion By-products
CFA	Coal Fly Ash
CFD	Cyclone Filler Dust
CKD	Cement Kiln Dust
CLSM	Controlled Low Strength Materials
CS	Compressive Strength
C-S-H	Calcium Silicate Hydrate
CQF	Cement-stabilised Quarry Fines
DMRB	Design Manual for Roads and Bridges
DoE	Design of Experiment
Dti	Department for Trade and Industry
EA	Environment Agency
EAF	Electric Arc Furnace
EIO – LCA	Economic Input Out – Life Cycle Assessment
EMV	Equivalent Mortar Volume
EU	European Union

EVD	Extreme Vertices Design
FA	Fly Ash
FABM	Fly Ash Bound Mixture
FGD	Flue Gas Desulphurisation
GBS	Granulated Blastfurnace Slag
GGBF	Ground Granulated Blast Furnace Slag
GGBFS	Ground Granulated Blast Furnace Slag
GGBS	Ground Granulated Blast Furnace Slag
GVA	Gross Value Added
HBBM	Hydraulically Bound Base Material
НВСМ	Hydraulically Bound Cement Mixtures
HE	Highways England
HMA	Hot Mixed Asphalt
HSC	High Strength Concrete
IBA	Incinerator Bottom Ash
IBAA	Incinerator Bottom Ash Aggregates
InDOT	Indiana Department of Transportation
ITZ	Interfacial Transition Zone
LHT	Large Hydrating Tailing
LKD	Lime Kiln Dust
LS	Limestone
l/s	Liquid Solid Ratio
MCHW	Manual of Contract Documents for Highway Works
MHT	Medium Hydrating Tailing
MK	Metakaolin
MPA	Mineral Products Association
MRA	Mixed Recycled Aggregates
Mt	Million tonnes
NG	Notes for Guidance
NHS	National Health Service
OECD	Organisation for Economic Co-operation and Development
ONS	Office for National Statistics
OPC	Ordinary Portland Cement
PC	Portland Cement
PCC	Portland Cement Concrete
PFA	Pulverised Fuel Ash
PG	Plasterboard Gypsum

PPG	Phosphogypsum
QD	Quarry Dust
QL	Quick Lime
QW	Quarry Waste
QWD	Quarry Waste Dust
RA	Recycled Aggregates
RAC	Recycled Aggregate Concrete
RAC - WB	Recycled Aggregate Concrete with Warwickshire Blend
RAF	Reclaimed Asphalt Filler
RAP	Reclaimed Asphalt Filler
RCA	Recycled Coarse Aggregates
RCA	Recycled Concrete Aggregates
RG	Red Gypsum
RMC	Recovery Mineral Components
RMRC	Recycled Materials Resource Centre
RMSWM	Recycled Materials and Secondary Waste Minerals
RSWM	Recycled and Secondary Waste Minerals
SCM	Supplementary Cementitious Materials
SBM	Slag Bound Mixture
SEM	Scanning Electron Microscope
SF	Silica Fume
SME	Small and Medium – Sized Enterprises
SS	Steel Slag
SSD	Steel Slag Dust
TRL	Transport Research Laboratory
TxDOT	Texas Department of Transportation
UCS	Unconfined Compressive Strength
UK	United Kingdom
UN	United Nations
UoW	University of Warwick
US	United States
USA	United States of America
USEPA	United States Environmental Protection Agency
WA	Water Absorption
WB	Warwickshire Blend
w/c	Water Cement Ratio
WCED	World Commission on Environment and Development

WMA	Warm Mixed Asphalt
WRAP	Waste and Resource Action Programme
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

CONFERENCES AND PAPERS PUBLISHED

List of Publications:

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List of Journal Papers 'Under Review'

- Bai Kamara, K. B., Ganjian, E. and Khorami, M., The use of waste gypsums, reclaimed asphalt filler and GGBS as full replacement of cement in road base – ASCE Journal of Materials in Civil Engineering. (Initial Date Submitted 27th October 2019 – Manuscript Number: MTENG – 9962).
- Bai Kamara, K. B., Ganjian, E. and Khorami, M., The effect of quarry waste dust and reclaimed asphalt filler in hydraulically bound mixtures containing plasterboard gypsum and GGBS – Journal of Cleaner Production. (Initial Date Submitted 29th October 2019 – Manuscript Number: JCLEPRO – D – 19 - 16386).

ABSTRACT

As the world is becoming aware of climate change and, as environmental policies and public concerns over the manufacture of cement and the extraction of our natural resources intensify, there is an increasing pressure and desperate need to use alternative materials to replace cement and primary aggregates. Environmental concerns over our desires for using products that are manufactured from the continuous extraction of our natural resources has necessitated a growing use of secondary waste minerals. In this research, comprehensive laboratory experiments were carried out to investigate the suitability of raw industrial by-products to be used as cementitious materials. The raw industrial by-products used in this research were: PG, V-B5G, RAF, QWD, BPD-C, BPD-H, MHT, SSD and GGBS. The by-products were obtained within the United Kingdom and predominantly sourced locally to develop a hydraulically bound cementitious material for applications in road (base), foundation and sub-grade in pavement construction for full replacement of Portland Cement.

Initial, material assessments were carried out based on X-ray diffraction (X-RD) and X-ray fluorescence (X-RF). High pozzolanic and / or cementitious properties were two important factors that were focused on for suitability of the raw materials in the research study. A broad range of laboratory tests were carried out to identify mechanical stability of the by-product binders and performance determined in strength development by time in terms of durability. High pressure flow tests were performed to determine the permeability of the materials and frost susceptibility tests were conducted to determine the freeze / thaw resistance of the materials. Absorption tests were carried out to determine the sorptivity of water by sustainable concrete made of by-products as binders and fines / coarse aggregates from recycled concrete. A statistical mixture design programme – Minitab 18, was used for the Design of Experiment (DoE). The Extreme Vertices Design (EVD) method was used to set the boundaries of the components in each group for the designs. Due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design. The effects of the combination of two / three mixes in the designs were considered in the analysis and discussions.

Eleven combinations of ternary binders referred to as 'Groups' were designed. There were thirteen mixes in each group. The first set (Groups 1 to 4) focused on the effects of MHT, RAF, BPD-C and BPD-H on mixes containing PG and GGBS. The second set (Groups 5 to 8) focused on the effects of MHT, RAF, BPD-C and BPD-H on mixes containing V-B5G and SSD. The third set (Groups 9 and 9A) concentrated on the effects of PG and V-B5G on mixes containing QWD and GGBS. Group 2A was added to compare the mechanical performance

of the two gypsums used in the research, using the same mix design in mixes containing RAF and GGBS. Their results were analysed, and the preferred groups optimised to obtain the best mix known as 'Warwickshire Blend.' Most of the groups used the same water content. Specimens in the phase one laboratory tests were evaluated on Compressive Strength at 7, 14, 28 and 90 days curing. Groups 2, 2A and 9 were identified as the three top groups. Long-term durability studies were carried out on selected mixes from the three top groups to determine the novel blend.

It was discovered that strength development on the hydraulic pastes was slow during the early stages of hydration for mixtures containing 40 - 60% GGBS. After 28 days and up to 90 days when the ultimate strength (i.e. 90 days age) of the hydraulic paste is achieved, strength increases with the presence of GGBS of up to 60%. Mixtures with the proportions of 20% PG (Plasterboard Gypsum), 20% RAF (Reclaimed Asphalt Filler) and 60% GGBS for Group 2; 10% V-B5G (Vitamin B5 Gypsum), 30% RAF and 60% GGBS for Group 2A; and 10% PG, 30% QWD (Quarry Waste Dust), and 60% GGBS for Group 9 attained the highest compressive strengths of 41MPa, 40MPa and 38MPa respectively at 90 days. Not only did these mixes performed exceptionally well on compressive strength, their results on durability tests satisfied relevant standard tests. One of the dominant factors that influenced the strength of the mixes in Groups 2, 2A and 9 was the presence of calcium sulfate hydrate - (CaSO₄2H₂O) in the PG, calcium sulfate - CaSO₄ (CaO + SO₃) in the V-B5G materials, calcium silicate – $CaSiO_3$ (CaO + SiO_2) in the GGBS and the pozzolanic activity (SiO_2 + Fe₂O₃ + Al₂O₃) in the RAF and QWD. The results suggest that all the eleven groups used in the research study, have mixes suitable for use as hydraulically bound concrete road construction materials. Mix 13 of Group 9 with the proportions of 10% PG, 30% QWD and 60% GGBS was identified as the 'Warwickshire Blend.' The contribution to knowledge for this research was the use of two waste gypsums (PG and V-B5G) and two guarry by-products (RAF and QWD) in combination with GGBS as full cement replacement in road (base) and foundation.

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CHAPTER 1 - INTRODUCTION

1.1 Introduction

Concerns about the depletion of natural resources and the effect that meeting the demand for aggregates may have on the environment has increasingly focused attention on the possibility of finding alternatives to naturally occurring materials as mentioned by (Sherwood 2001). In this research, an attempt was made to investigate some new replacement materials for cementitious binders. This research looked at the potential use of nine industrial by-products as alternative to cement. Two different types of factory gypsum wastes: Vitamin B-5 Gypsum (V-B5G) and Plasterboard Gypsum (PG) were used to activate three pozzolanic materials: i) Ground Granulated Blast Furnace Slag (GGBS); ii) Quarry Waste Dust (QWD); and iii) Reclaimed Asphalt Filler (RAF), combined with Bypass Dust (BPD-C and BPD-H), Medium Hydrate Tailing (MHT) and Steel Slag Dust (SSD) in different proportions as binary and ternary semi-dry pastes. Some of these materials have been identified as having cementitious and pozzolanic properties and as an alternative to cement. These various types of by-products were assessed to have potential for use in road (base) and road foundation. Recycled concrete was used as fine and coarse aggregate with the novel paste to create sustainable concrete. The new replacement materials will reduce the impact the extraction of naturally occurring materials have on the environment.

Previous research studies have been carried out to explore the partial replacement of cement using different industrial by-product materials in the construction industry. Unlike previous studies investigated by (Limbachiya, Ganjian and Claisse 2015; Raghavendra et al. 2016; Tan, Doh and Chin 2017), this research study draws attention to the <u>full replacement of cement</u> in concrete.

1.2 Background Information

The cement industry is one of the biggest waste producer industries in the world (Modarres, Ramyar and Ayar 2015). Approximately, 7% of the global carbon dioxide emissions come from the manufacture of cement (Andrew 2018; Benhelal et al. 2013; Li et al. 2013; Meyer 2009; Siddique and Rajor 2012). Portland cement itself is ecologically harmful in that the production of one tonne of cement equates to approximately one tonne of carbon dioxide being expelled into the atmosphere (Pourkhorshidi et al. 2010). The

production of Portland cement, an essential constituent of concrete, leads to not only the release of significant amount of global CO₂ emissions, but also other harmful greenhouse gases (Naik 2007). The construction industry is identified by the UK Green Building Council as one of the most emission-intensive industries, accounting for approximately 50% of greenhouse gas production in the UK (Dadhich et al. 2015). There is no doubt an increasing demand for the use of industrial by-products and recycled materials due to the stress on our natural resources and environmental systems. The UK Mineral Production Association (MPA 2016) in the New WRAP Guidance on the Quality Protocol requirements to produce aggregates from inert waste, notes that 28% of Great Britain aggregates market was supplied by recycled and secondary waste minerals in 2015, three times higher than the average European market share.

Waste management and the use of industrial by-products are important aspects of government policies around the world aimed at re-enforcing current trends regarding the conservation of our natural resources. Recent increase in the use of environmental controls meant that some of these wastes are recovered within the manufacturing process. Most of the wastes however, are removed and disposed of to landfill sites. The increasing shortage of land available for waste disposal, the penalties introduced by local authorities in disposing of waste, and the increased manufacturing of by-products, it is inevitable that increased use of these industrial by-products be explored in pavement and other construction applications to help prevent depletion of virgin materials which is accompanied by environmental degradation and lead to economic problems.

1.2.1 The World on Climate Change

Ambitious objectives in the United Nations (UN) Climate Change Conference in Paris 2015 to reduce emissions by 40% to 70% (scenario to achieve 2°C) or by 70% to 95% (scenario for 1.5°C) by 2050 compared to 2010, or the achievement of "zero emissions" by the middle of the century was tabled in the summit.

One of the points that remained in suspense in the Paris UN Climate Change Conference was the ratification of an ambitious agreement to limit the temperature rise to 2°C. This would require limiting greenhouse gas emissions through energy efficiency measures, amongst other things. In November 2016 at the UN Climate Change Conference in Marrakech, nations have agreed on the Paris Climate Agreement. The treaty commits governments to take actions to keep global temperatures from rising by 2°C above pre-industrial levels and to do their best to keep that rise to less than 1.5 degrees (McGrath 2016).

Statement of the American Meteorological Society (AMS) in August 2012 on climate change. "It is clear from extensive scientific evidence that the dominant cause of the rapid change in climate of the past half century is human – induced increases in the amount of atmospheric greenhouse gases, including carbon dioxide (CO₂), chlorofluorocarbons, methane, and nitrous oxide. The most important of these over the long term is CO₂, whose concentration in the atmosphere is rising principally as a result of fossil-fuel combustion and deforestation."

1.2.2 UK on Climate Change

The original target for Climate Change Mitigation in the UK has now been revised in the Climate Change Act to be an 80% (previous 60%) cut in greenhouse gas emissions by 2050 on the 1990 levels, and 34% by 2020 (HM Government 2009). The UK Government's ambition to reduce emissions by 2050 is much greater than that set by the UN by 10%.

The UK Government is very keen to promote Sustainable Development. Many Local Authorities and Government Agencies are working towards the same objectives in the construction projects that they procure. To help promote the reform throughout the industry including the private sector, the Department for Trade and Industry (Dti) has sponsored a number of new programmes to help educate civil engineers, architects and builders about new technologies, materials and methods available to help with the smooth implementation of new legislation. Some of these organisations include the Waste and Resource Action Programme (WRAP), Viridis, Sustainable Construction and CIRIA. These organisations consult regularly with practicing professionals and work closely with the Environment Agency (EA) and the Transport Research Laboratory (TRL) to ensure their guidance is accurate and up-to-date.

(Rhodes 2015), states that in 2014, the construction industry's output was £103 billion, 6.5% of the total UK economy. The industry's output rose in 2014 by 9.5% in real terms, the biggest increase since at least 1990.

One of the four aspirations set by the Coalition Government in Construction 2025 (July 2013), is for the UK Government to work in conjunction with a range of industry bodies to achieve ambitions that include a 50% reduction in greenhouse gas emissions in the built environment.

(Construction 2025), went on further to outline an "action plan" which includes the steps the Government and industry will take in the short and medium term to achieve low carbon and sustainable construction. This includes using technological developments and new materials to reduce the ecological impact of construction.

The ambitious target to reduce the overall carbon emissions by 50% at 2025 and the role of the construction sector within this target cannot be overemphasised given that it has been estimated that construction can potentially influence 47% of the total UK carbon emissions (Dadhich et al. 2015).

1.2.3 Sustainable Development

(Borowy 2014) used the classic definition of Sustainable Development published by the World Commission on Environment and Development (WCED) in 1987, also referred to as the Brundtland Report - *Our Common Future* as "development that meets the needs and aspirations of the present generation without compromising the ability of future generations to meet their own needs".

1.3 Statistics on Construction Waste, Recycled and Secondary Waste Aggregates

(Directive 2008) requires that waste be managed without endangering human health and harming the environment, and without risk to water, air, soil, plant or animals, without causing a nuisance through noise or odours, and without adversely affecting the countryside or places of special interest.

1.3.1 Statistics on Construction Waste

The UK generated 222.9 million tonnes of total waste in 2016. Over half (61%) of this was generated by construction, demolition and excavation (CD&E) activities. The recovery rate from non-hazardous construction, demolition and excavation waste in the UK in 2016 was 91%. This is more than the EU target for the UK to recover at least 70% of this type of waste by 2020. (UK Statistics on Waste 2019).

1.3.2 Statistics on Recycled and Secondary Waste Aggregates

MPA 2016, states the use of recycled materials and secondary waste minerals in Great Britain aggregate market, which for the year to 31st December 2015 was 225 million tonnes (Mt), has increased rapidly rising from 30 Mt per annum in 1990 to over 63 Mt in 2015. Over that period the share of the aggregate market supplied from recycled and secondary sources has risen from 10% to 28%.

When compared to an increase of primary aggregates production in Great Britain of 10 Mt between 2012 and 2013, 1 Mt of recycled materials and secondary waste minerals in the same period was produced. Recycled materials and secondary waste minerals (RMSWM) therefore accounts for 28% of aggregates sold in Great Britain in 2013 based on 143 Mt of primary aggregates and 55 Mt of RMSWM. (MPA Report, 2014).

A study carried out by the waste and resources action programme (WRAP) estimates that construction, demolition and excavation waste production alone accounts for 120 Mt per annum in the UK (Saride, Puppala and Williammee 2010). Approximately 70 Mt of construction and demolition waste is deposited to landfill sites every year. Around 15% of these recycled / secondary materials are being used in road construction in the UK (Lambert, Fleming and Frost 2006).

These statistics show that there has not been much change in Great Britain to the market share of RMSWM in the last decade.

1.4 Challenges and Benefits of the use of Secondary Aggregates and Recycled Aggregates

1.4.1 What are Secondary Aggregates

Secondary aggregates are defined by the British Geological Survey as:

- aggregates obtained as a by-product of other quarrying and mining operations, such as china clay waste, slate waste and colliery spoil; or
- aggregates obtained as a by-product of other industrial processes, such as blast furnace / steel slag, coal-fired power station ash, incinerator ash and spent foundry sand.

Secondary aggregate sometimes referred to as by-product is defined in Article 5 of (Directive 2008). Provisions of the article were based on the case law of the Court of Justice of the European Union (EU). By-product is a substance or object, resulting from a production process, the primary aim of which is not the production of that item, may be regarded as not being waste referred to in point (1) of Article 3 but as being a by-product only if the following conditions are met:

- where further use of the substance or object is certain:
- where the substance or object can be used directly without any further processing other than normal industrial practice;
- where the substance or object is produced as an integral part of a production process; and
- where no further use is lawful, the substance or object fulfils all relevant product, environmental and health protection requirements for the specific use and not lead to overall adverse environmental or human health impacts

A much simple definition of by-product was carried out in a report prepared by an Organisation for Economic Co-operation and Development Road Research Group (OECD 1977). The Group defined by-product as a material that is obtained during the production of another material which is considered to be the main product.

1.4.2 What are Recycled Aggregates

These are wastes that arise from different sources, they include the following:

- construction and demolition of buildings and structures;
- planings from existing concrete and bituminous pavement; and
- aggregates derived from crushed railway track ballast

1.4.3 Challenges to the use of Recycled and Secondary Waste Minerals

In January 2006, WRAP commissioned a study "The Promotion of the benefits of Recycled and Secondary Aggregates to Small and Medium – sized Enterprises (SME) Sector of the Construction Industry". The study revealed obstacles to the use of recycled and secondary waste minerals (RSWM) by SME's. Summary of some of the obstacles highlighted in the study are as follows:

- the client lack of interest in the materials hence reluctant to specify in tender documents;
- the view on the quality of RSWM. Many respondents felt that within the industry, RSWM was widely and incorrectly regarded as of inferior quality to primary aggregates;
- the lack of confidence with materials of adequate technical specifications degrades it credibility;
- the avoidance of risks Architects, Civil Engineers and Structural Engineers to specify in project design; and
- the attitude of decision makers (site managers), despite their organisation supporting and promoting the use of RSWM.

One of the challenges to the use of RSWM is the reluctance by the major players or drivers in the UK construction industry. Highways England (HE) and other highway authorities such as city and county councils are one of the main contributors to the industry, hence very reluctant to exert a considerable influence on the use of these materials at national level. Transport of materials remains one of the most inhibiting factors in the use of RSWM. The relationship between the quantities of materials required against the distance travelled to the point of delivery in regard to haulage costs remains a major factor influencing the wider use of these materials.

Based on the requirements of codes and national highway design guides, the main mechanical performance criteria for hydraulically bound mixtures and cement bound granular mixtures made from recycled and secondary waste minerals is compressive strength. One of the limitations in existing national standards is the lack of laboratory testing requirements to understand the long-term durability performance of these materials.

1.4.4 Benefits to the use of Recycled and Secondary Waste Minerals

Based on the three-pillar approach to sustainable development which involves the integration of economic, environmental and social elements, the benefits to the use of RSWM can be summarised as follows:

- Economic where sources of these materials are produced close to centres of demand, its economic benefits include reducing haulage journeys thereby cutting the costs of transporting virgin aggregates. There is a business case for cost savings over primary or new aggregates and easing of traffic congestion in the network will also help the local and regional economy.
- Environmental its environmental benefits include eliminating altogether the demand for primary aggregates, thereby conserving our natural resources, decreasing energy consumption, diverting waste from landfill sites and most of all increasing carbon dioxide savings through lower transport distances from locally available RSWM. Locally sourced RSWM will help shorten the timescales associated with some construction techniques.
- Social its social benefits include creating new jobs in the local area, reducing haulage activities thereby reducing congestion, increasing road safety and cutting air and noise pollution.

WRAP 2005, case studies on a number of highway schemes in the UK demonstrated that financial savings can be obtained from the use of RSWM. One of the schemes mentioned was the Lichfield to Cannock, Burntwood Bypass in Staffordshire in 2001 where the use of incinerator bottom ash as aggregates with coal fly ash as part of the binder was used in the sub-base and base. A direct total cost savings of approximately £63,000 was made in delivering this scheme using secondary waste minerals as alternatives to primary aggregates.

1.5 Aim and Objectives of Study

1.5.1 Aim

This research study is to use by-products and recycled materials in order to reduce the impact the extraction of naturally occurring materials have on the environment. This research investigates the potential use of vitamin B5 gypsum (V-B5G) and plasterboard gypsum (PG) combined in mixtures with by-pass dust (BPD), medium hydrate tailing (MHT), reclaimed asphalt filler (RAF), quarry waste dust (QWD), ground granulated blast furnace slag (GGBS) and steel slag dust (SSD) to investigate a novel binder with pozzolanic / cementitious properties to fully replace cement. The final aim is to design a sustainable concrete mix from the novel paste using recycled concrete and / or natural aggregates that can be used as road base.

1.5.2 Objectives

Optimisation of the by-products and recycled materials in a novel mix for mechanical stability, physical properties and chemical composition in road pavement will be the main theme of this research study. Other objectives can be broken down into the following:

- to identify industrial waste minerals that are being sent to landfill sites in the United Kingdom and to investigate their feasibility in road pavement construction;
- the innovative use of a broad range of different industrial by-products with cementitious properties;
- the use of a range of different quarry waste with pozzolanic properties;
- to partially replace cement with industrial by-products as main constituent of the binder in the novel mix;
- to replace primary or virgin aggregates with recycled concrete as fine and coarse aggregate in the sustainable concrete;
- the identification and evaluation of key barriers to sustainable resource; and
- the recommendations in areas where further studies can be explored to complement the outcome of this research

1.5.3 The Research Questions

The research questions are:

- can full replacement of cement be achievable with required properties?
- would the use of secondary waste minerals or the combination of secondary waste minerals and recycled materials be a potential replacement of existing Hydraulically Bound Concrete Mixtures (HBCM)?
- would the use of secondary waste minerals be a potential treatment of the subgrade as the capping and sub-base layer;
- would the adoption of these construction practices encourage the use of more sustainable highway construction? and
- would this construction method avoid the need to extract natural resources, improve environmental goals and offer value for money?

1.5.4 Research Gap and Novelty

The novelty of this research study is to close the knowledge gap in the use of gypsum wastes and quarry by-product. The combination of these waste minerals together with those mentioned in Section 1.5.1 can be used as binders for road base, and on sub-grade as soil stabilisation. The novel binder together with recycled concrete and / or natural

minerals as fines and coarse aggregates, is a fully sustainable cement bound granular mixture that can be used in pavement engineering.

CHAPTER 2 - METHODOLOGY

2.1 Methodology Overview

The outline of the methodology that has been followed in this research study is represented in Figure 2.1 below:



Figure 2.1: Outline Methodology

This research is summarised in five parts:

- Literature Review on industrial by-products, particularly those similar to the ones used in this research study were carried out;
- Identifying suitable industrial by-products mentioned in Section 2.3 of this report;

- Experiments on suitability of by-products identified. Some of the experiments include carrying out XRD and XRF to determine the mineralogical properties, chemical compounds and chemical compositions of the materials obtained. Other experiments to determine whether the by-products were pozzolanic and / or cementitious were carried out;
- Experiments on materials with potential were carried out. Some of these
 experiments include compressive strength tests, particle size distribution tests,
 high pressure permeability tests, freeze-thaw tests, and tests to determine the
 absorption properties of the preferred blends; and
- The analysis method used was a statistical mixture design programme "Minitab 18." Eleven combinations of ternary binders referred to as 'Groups' were designed. Results from the experiments were evaluated against data from similar research studies. Interpretation and discussions of the finding were carried out.

2.1.1 Report Structure

The report continues with Chapter 3.0 covering literature review of previous research on industrial by-products, particularly on waste materials identified to be used in this research study.

Chapter 4.0 covers laboratory experiments to determine the suitability for use in this research study of the industrial by-products obtained from various sectors.

Chapter 5.0 details the Phase One experimental programme used in this research study. It details the experimental strategy and testing procedures used.

Details of the Phase One mix designs and Phase Two (identifying the chosen groups) experiments in this research study are explained in Chapter 6.0.

Chapter 7.0 covers the analysis and discussions of the laboratory test results obtained from the Phase One and Phase Two experiments.

Phase Three deals with the results of further experimental tests carried out to determine the optimum blend from the chosen Groups and to firm-up a mix design for this research study (Chapter 8.0).

Chapter 9.0 covers Phase Four experimental tests on sustainable concrete made from the novel 'Warwickshire Blend' and Chapter 10 covers the conclusions. The sustainability measures, applications of research materials and recommendations for further research are explained in Chapter 11.0.

2.2 Initial Research in Identifying Materials for Study

2.2.1 Initial Primary Research

The initial primary research was conducted in a form of telephone interview, an attempt to gain an overview of new waste materials that have emerged in various sectors of the industry in the UK was carried out. The interview was focused in identifying suitable waste minerals that have been obtained as by-products derived from industrial processes and are currently being disposed of to landfill sites.

Minerals and recycling companies that supply both naturally occurring primary aggregates, recycled and secondary waste aggregates were approached.

The interview through telephone conversation did not reveal the outcome expected. This method of seeking valuable information was therefore abandoned and a second approach based on the primary data was considered.

2.2.2 Initial Secondary Research

The initial secondary research was conducted through email correspondence. Emails were sent to senior managers of companies in order to gain an understanding of available waste minerals that are currently being sent to landfill sites for disposal.

Compared to the initial primary research, interests were expressed and replies were received from some of the companies. Companies were expressing interests in the use of alternative materials as substitute for materials that are traditionally used in pavement construction. The companies that replied were very keen on the research study and expressed environmental concerns associated with disposing of these waste minerals to landfill sites. They were also drawn to the idea of maximising the use of recycled and secondary waste minerals and the thoughts of the research study adhering to the principles of sustainable development which requires the prudent use of our natural resources and maximum use of recycled construction materials where appropriate.

2.3 Identifying Materials for the Research Study

The following sectors were approached:

- Casting Federation;
- Sugar Factory;
- Aggregates and Quarry Industry;
- Cement Factory;
- Chemical Industry;
- Steel Industry; and
- Mineral Aggregates and Recycling Centres

Approximately over 60% of the sectors listed above showed interest in the research study. The nature of questions to the companies can be found in Appendix A. The companies who responded were involved in the following sectors:

- Aggregates and Quarry Industry;
- Cement Factory;
- Chemical Industry;
- Steel Industry; and
- Mineral Aggregates and Recycling Centres

Appendix B show copies of correspondence made to the companies via their website. Copy of emails response correspondence from companies can be found in Appendix C. Copy of emails correspondence in Appendix D illustrates the author's response to the companies replies. Summary of the materials supplied from the companies who responded are listed in Table 2.1.

Sector	Materials Supplied	
Aggregate and Quarry Industry	Reclaimed Asphalt Filler (RAF)	
	Quarry Waste Dust (QWD)	
	Lime Kiln Dust (LKD)	
	Medium Hydrated Tailing (MHT)	
	Large Hydrated Tailing (LHT)	
Cement Industry	By-pass Dust from Cemex (BPD-C)	
	By-pass Dust from Hanson (BPD-H)	
Chemical Industry	Vitamin B5 Gypsum (V-B5G)	
Steel Industry	Steel Slag Dust (SSD)	
Mineral & Aggregate Recycling		
Plasterboard Company	Plasterboard Gypsum (PG)	
Day Aggregates	Type 1 Recycled Concrete	
	Type 1 Eco Blend	
	Incinerator Bottom Ash	
KSD Aggregate Limited	Type 1 Recycled Sub-base	
	Incinerator Bottom Ash (various types)	
	Recycled Capping 6F5	
Raymond Brown Minerals	Recycled All-in Ballast	
	Recycled Type 1	
	Recycled Glass	
	Recycled Grit	
	Recycled Clay	

Table 2.1 Summary of sectors replied, and materials supplied

2.3.1 Aggregates and Quarry Industry

Tarmac OCR identified Reclaimed Asphalt Filler (RAF), Quarry Waste Dust (QWD), Lime Kiln Dust (LKD), Medium Hydrated Tailing (MHT) and Large Hydrated Tailing (LHT) as waste minerals. These materials have very low commercial value. Tarmac believed that the research study would benefit in exploring the use of these materials as an alternative to virgin materials.

Both the RAF and QWD were sourced locally from their Mancetter Quarry in Atherstone, North Warwickshire. The RAF material is produced by their Via Nova Asphalt Plant and conveyed to the plant reclaimed duct silo. The material is formed during the processing of asphalt using sedimentary rocks. The QWD can also be referred to as Cyclone Filler Dust (CFD). The QWD is a waste material, produced as a by-product of the screening and crushing processes. Approximately 80kg each of these samples were obtained for the initial study.



Figure 2.2: Tarmac Mancetter Quarry - Atherstone, Warwickshire, 2017

The lime products were not sourced within the West Midlands. The samples came from their Tunstead - Old Moor Quarry complex, near Buxton that links the Derbyshire – Peak District National Park boundary. Approximately 70kg each of these samples were delivered to Coventry University.

The LKD has calcium carbonate (CaCO₃) content of 80% and calcium oxide (CaO) of 20%. The material is collected from air emission abatement plant (bag filters) that captures particulate material. The MHT has (CaCO₃) content 20% and calcium hydroxide (Ca(OH)₂) of 80%. The hydrate products are graded and medium pieces are removed from the final product. The CaCO₃ and Ca(OH)₂ content on the LHT is the reverse of the MHT product with 80% of CaCO₃ and 20% of Ca(OH)₂. The hydrate products are graded and large pieces are removed from the final product.



Figure 2.3: Tarmac Tunstead – Old Moor Quarry Complex, 2017

2.3.2 Cement Factory

The two cement factories (Cemex and Hanson-Castle Cement) that responded identified By-pass Dust (BPD) as the main waste that they produce. Both companies, together produce approximately 30,000 tonnes of BPD per year.

Approximately 80kg of BPD was obtained for initial experimental study from each of the cement factories. The Cemex sample was sourced from the Rugby Plant in Warwickshire and the Hanson sample came from the Warwickshire / Leicestershire boundary.



Figure 2.4: Cemex Rugby Cement Plant – Rugby, Warwickshire, 2012

2.3.3 Chemical Industry

The vitamin B5 gypsum (V-B5G) was sourced from Scotland. Approximately 90kg of the sample was delivered to the university. The material, when delivered had a moisture content of approximately 40%. V-B5G is produced by the pharmaceutical industry. This industrial waste is produced from the manufacture of vitamin B5 (Pantothenic acid). The

acid waste stream which is a by-product from the manufacture of vitamin B5 arises from precipitation of calcium sulphate which reacts with calcium hydroxide to form calcium sulphate gypsum.

The vitamin B5 can be manufactured as medicinal drugs and as ingredients or supplement in foods.

Approximately 10,000 tonnes of this waste material is produced by the company each year. The waste material contains approximately 128 mg/kg of aluminium, 173,912 mg/kg of calcium and 128,349 mg/kg of sulphur. Other chemical properties include ammoniacal nitrogen (329 mg/kg) and ammonia – water soluble (321 mg/kg) and the material is white in colour.

2.3.4 Steel Industry

The steel slag dust (SSD) was sourced from Tarmac's Port Talbot site in Wales. Approximately 80kg of this sample was obtained for the initial study. A further request was made for 100kg to be delivered to the university.



Figure 2.5: Tarmac Port Talbot Quarry – Wales, 2014

2.3.5 Minerals and Aggregate Recycling Centres

All the companies that responded have supplied samples for the initial experimental study:

- Day Aggregates;
- KSD Recycled Aggregates Limited;
- Raymond Brown Minerals and Recycling Limited; and
- Barratt Homes

2.3.5.1 Day Aggregates

The samples obtained from Day Aggregates were:

- Type 1 Recycled Concrete (50mm crushed concrete);
- Type 1 Eco Blend mixture of Primary and Recycled Aggregates; and
- Incinerator Bottom Ash (IBA) Sand 0 4mm

The Type 1 Recycled Concrete and Type 1 Eco Blend were sourced from Croydon, London. The IBA was sourced from Brentford, London.

2.3.5.2 KSD Recycled Aggregates Limited

The samples obtained from KSD Recycled Aggregates Limited were:

- Type 1 Recycled Sub-base;
- Incinerator Bottom Ash (IBA) Washed Sand 0 4mm;
- Incinerator Bottom Ash (IBA) Pressed IBA 0 0.063mm;
- Incinerator Bottom Ash (IBA) IBA Aggregates 4 -10mm; and
- Recycled Capping 6F5– Clean Crushed Concrete, Brick and Bituminous Materials 75mm

The IBA from KSD was locally sourced from a domestic waste incinerator in Coventry UK. The municipal household waste derived from three highway authorities in the West Midlands namely: Coventry, Solihull and Warwickshire. In total, approximately 200kg of these samples were obtained.

2.3.5.3 Raymond Brown Minerals and Recycling Limited

Recycled Incinerator Bottom Ash Aggregates (IBBA) Sub-base was obtained from Raymond Brown Minerals for the initial experiment. The samples obtained were sourced from two sites namely: Yeardley and A303 Hampshire.

Additional five different samples were received from the company:

- Recycled All in Ballast 20mm;
- Recycled Type 1 (Crushed Concrete suitable for use in capping layer and subbase);
- Recycled Glass;
- Recycled Grit; and
- Recycled Clay

The total materials received from the company was approximately 150kg.

2.3.5.4 Plasterboard Company

The plasterboard waste was collected from a housing development site in Nuneaton, North Warwickshire. The plasterboard waste arose from off-cuts and damaged boards and the project involved the construction of houses by one of the leading housing developers – Barratt Homes. Approximately, 150kg of the sample was obtained.

2.3.6 Analysis of Data from Initial Secondary Research

Analysis of data from the initial secondary research was mainly based on sectors / companies with potentials to produce binders as industrial by-products. One question was asked to all the participants. The question was in four parts: Q1(i); Q1(ii); Q1(iii); and Q1(iv).

Section Q1(i) of the questionnaire required the companies to confirm whether they produce industrial waste minerals during the manufacturing process of the main products. Section Q1(ii) asked whether the waste minerals produced were being taken to landfill

sites for disposal. Section Q1(iii) gauged the participants approach to environmental policies and controls in regard to recovery all, some or none of the secondary waste minerals within the manufacturing process. When some or none of the waste minerals were used within the manufacturing process of the main products, Section Q1(iv) asked the participants whether it would be possible to provide samples that could be used to investigate the materials' mechanical performance in pavement engineering.

Figure 2.6 show percentages apportioned for binder materials obtained by sectors. It can be seen from the graph that approximately 50% of the binder materials were obtained from the aggregate and quarry industry. The second highest was from the cement industry (approximately 20%). The remaining three industries had equal share of approximately 10%.



Figure 2.6: Binder materials obtained by sectors

Figure 2.7 show percentages apportioned for binder materials by companies. Approximately, 60% of the binder materials obtained came from Tarmac OCR. All the remaining four companies shared approximately 10% each.



Figure 2.7: Binder materials obtained by companies

Analysis of Section Q1(i) of the questionnaire showed that the companies from all the sectors that responded confirmed they produce industrial waste minerals during the manufacturing process of the main products. Regard to the response received for Section Q1(ii), not all of the waste minerals produced were taken to landfill sites. Even though some of the waste minerals are recovered within the manufacturing process as indicated from all off the respondents, most are disposed of to landfill sites. The response to Section Q1(iii) was the same to that of Section Q1(ii) in respect to recovering some off the wastes thereby adhering to environmental policies and controls. All the respondents for Section Q1(iv) were able to provide the materials that were used in the research study.

CHAPTER 3 - LITERATURE REVIEW

3.1 Cement Replacement Materials in Concrete Pavement

Some by-product materials have potential for reuse in road construction as cement replacement materials. Potential uses are summarised in Table 3.1, a modified version of the table produced by (Sherwood 1995).

	Potential uses		
Material	Unbound Capping layer	Unbound Sub-base	Cement-bound Material
China clay sand	High	Some	High
Demolition debris (crushed concrete)	High	High	High
Blast Furnace Slag	High	High	High
BOS Slag	Some	Some	Low
EAF/refractory blend	Some	High	Low
Minestone spoil - burnt	High	Some	High
Minestone spoil - unburnt	Low	None	Some
Incinerator ash	Some	Some	None
Pulverised fuel ash	Low	Low	High
Slate quarry residues	High	High	Some
Spent oil shale	High	Some	High

Table 3.1 Summary of potential uses of by-product materials in concrete pavement (Baldwin et al. 1997)

Extensive research has been carried out by others and this differs from what has been stated in Table 3.1 for the potential use of BOS in cement-bound materials.

The compositional relationships between some of these by-products and cement are shown in Figure 3.1 and Table 3.2.



Figure 3.1: Compositional relationships between cement and cement replacement materials (Taylor 2002)

Туре	Silica content (per cent)	Lime content (per cent)	Relative density	Fineness	Particle shape
PFA	50	3	2.3	6% retained on 45 μm sieve	spherical
GGBFS	38	40	2.9	specific surface 400 m²/g	irregular
Microsilica	92	0.1	2.2	specific surface 15 000 m²/g	hollow or solid spheres

3.2 Potential use of by-product in Road Construction

Evidence from research studies carried out by (Ganjian, Essie et al. 2008), (Nataatmadja and Tan 2001), (Lepech and Li 2010), (Karami et al. 2013), (Aziz et al. 2014) and (Konsta-Gdoutos and Shah 2003) show that the performance of by-products can be enhanced

significantly in combination with either cement or other by-products. These by-products in some cases act as a complete cement replacement material. In other cases, due to their cementitious and pozzolanic properties, they act as activators to the binders and / or used as fillers in concrete products. It is a common practice in some countries to use by-product in road construction. Below are some examples on how by-products are used and how they can be improved by combination with either cement or other by-products:

- lime / cement, lime / fly ash, fly ash/ cement and lime / GGBS combinations used as a hydraulic binder;
- Pulverised Fuel Ash (PFA) used as a substitute for cement in concrete products;
- Blast Furnace Slag (BFS) used in base and sub-base construction;
- Colliery Shale used as bulk fill for embankment construction; and
- Cement Kiln Dust (CKD) and By-pass Dust (BPD) used as a potential substitute for cement in concrete pavements and concrete blocks

If the correct combination percentage is used, the materials can be optimised for mechanical stability.

3.3 Previous use of By-product in Hydraulically Bound Concrete Pavement

3.3.1 By-product use in Pavement in the United Kingdom

In the United Kingdom (UK), the Specification for Highway Works (MCHW 1) permits the use of a range of secondary waste and recycled materials. The use of these materials in pavements help reduce the environmental impact in the extraction of primary aggregates, reduce waste being sent to landfill sites and help stimulate the use of secondary minerals.

Table 3.3, a modified version of a table taken from a withdrawn highway design standard (DMRB HD35/04 2004) shows which by-products and recycled materials can be used in the sub-base layer and the base layer. The table details hydraulically bound mixtures only.

Table 3.3 Provisions for the Use of Secondary and Recycled Materials (DMRB HD35/04 2004)

Application and Series >	Hydraulically Bound Mixture for Sub-base and Base	
Material V	800	
Blast Furnace Slag	\checkmark	
Burnt Colliery Spoil	\checkmark	
China Clay Sand/Stent	\checkmark	
Coal Fly Ash/Pulverised		
Fuel Ash (CFA/PFA)	\checkmark	
Foundry Sand	\checkmark	
Furnace Bottom Ash (FBA)	\checkmark	
Incinerator Bottom		
Ash Aggregate (IBAA)	\checkmark	
Phosphoric Slag	\checkmark	
Recycled Aggregate	\checkmark	
Recycled Asphalt	\checkmark	
Recycled Concrete	\checkmark	
Recycled Glass	\checkmark	
Slate Aggregate	\checkmark	
Spent Oil Shale/Blaise	\checkmark	
Steel Slag	\checkmark	
Unburnt Colliery Spoil	\checkmark	

The tick (\checkmark) shows that the material is permitted as a constituent if the material complies with the Specification (MCHW 1). All the materials listed in the table can be used as hydraulically bound mixture (HBM) for sub-base and base.

(DMRB CD 226 2020) give examples of hydraulic bound base materials (HBBM). Examples of these materials are:

• Cement Bound Granular Mixture (CBGM);

- Slag Bound Mixtures (SBM); and
- Fly Ash Bound Mixture (FABM)

The laboratory mechanical performance categories or classifications are detailed in the Specification (SHW 2020a) MCHW Series 1000.

WRAP 2005, defined hydraulically bound materials as mixtures that set and harden by hydraulic reaction and that have a water content suitable for compaction by rolling. They include cement bound mixtures (CBM) based on the fast setting and hardening characteristics of Portland cement, as well as mixtures based on slow setting and hardening hydraulic binders such as pulverised fuel ash (PFA) and granulated blastfurnace slag (GBS).

TRL HTRL 404 report, case studied the use of Incinerator Bottom Ash (IBA). The study futures a 4km by-pass to Waltham Abbey which was designed originally in accordance with DMRB Volume 7. The design was altered to obtain environmental benefits. Clay sub-grade was stabilised to construct the sub-base. This totally replaced virgin aggregates in the production of the cement bound granulated mixtures (CBGM). The CBGM aggregate comprised 100 percent IBA and the compressive strength satisfied the design criteria.

(Reid and Chandler 2001), another case study came from the result of a UK government sponsored research project. The Echline project was constructed near Edinburg at South Queensferry. The objective was to determine what could be achieved using secondary waste and recycled materials in terms of minimising both energy consumption and virgin aggregates use. The scheme was designed to carry 100 msa over 40-year life. It is a single carriageway, 100 m long by 6 m wide.

The materials used in the construction were:

- Subgrade soft clay conditioned in-situ with lime and stabilized in-situ with PFA and cement
- Subbase spent oil shale aggregates bound with PFA and cement

- Base crushed construction and demolition waste bound with granulated blast furnace slag and PFA, activated with gypsum and lime
- Binder hot mix asphalt incorporating recycled asphalt planings and foundry sand
- Surface Course cold-lay bitumen emulsion asphalt incorporating steel slag coarse aggregate, blast furnace slag fine aggregate and polymer modified binder.
- 3.3.2 By-product use in Pavement in the European Union

Countries in Europe such as Denmark, Germany and the Netherlands have stringent policies in support of the use of secondary waste minerals in road construction. In general, by-products used in Europe are similar to those in the UK. These by-products are used in unbound sub-base / base, embankments, in bituminous bound layers, as concrete filler, in hydraulic bound layers and in earthworks.

TRL HTRL 404 report, states that 100 percent steel slag and coal bottom ash in Denmark is recycled and used in road construction. This gives a magnitude of 0.066 and 1.17 million tonnes respectively. For Germany, 100 percent blast furnace slag, with 9.2 million tonnes produced and used in road construction. The Netherlands however, a populous country with scarce natural resources has a 100 percent recycling rate in blast furnace slag, steel slag, coal bottom ash, coal fly ash and construction and demolition waste. This gives a magnitude of 1.32, 0.55, 0.09, 0.94 and 10.1 million tonnes respectively.

(Motz and Geiseler 2001) summarised experiences with the application of steel slags in road construction in Germany. Test roads were built more than 25 years ago to demonstrate steel slags advantageous technical properties. Steel slags were not only used for unbound layers such as sub-base and subgrade materials, they were also used for bound layers like the base and surfacing materials. The test roads were divided into sections which gave the possibility of investigating steel slags in comparison to naturally occurring aggregates such as crushed gravels. It was concluded that by building the test roads, the following results could be achieved:

 the rough surface of the processed steel slag aggregate provides a much stronger bearing capacity compared to using other aggregates;

- there is no evidence to show that heavy rainfall influence the bearing capacity of the unbound layers built with steel slag mixtures;
- the steel slag aggregate mixtures are permanently stable if the requirements for the volume stability have been fulfilled;
- the resistance to polishing of surface layers remain at a high level over a long-term period; and
- the roads built with steel slags as unbound and bound aggregates do not influence the environment by leaching.
- 3.3.3 By-product use in Pavement in the USA

The United Sates Environmental Protection Agency (USEPA) in conjunction with the U. S. Department of Transport and the U. S. Department of Energy carried out a study on increasing the usage of recovered mineral components (RMC) in highway construction projects. It was found in the report that the use of RMCs can reduce energy consumption and save costs on construction projects (EPA Report, 2008). It was identified in the study that the most significant materials beneficially used in terms of quantity were coal fly ash (CFA), blast furnace slag aggregates (BFSA) and flue gas desulphurisation (FGD) gypsum. Some of the issues the report addressed were:

- identifying potential mechanism to achieve greater substitution of RMC in cement, concrete or hydraulically bound mixtures;
- evaluating the feasibility of establishing standards for optimized substitution rates of RMC; and
- identifying potential environmental or economic effects that may result to greater substitution of RMC.

Table 3.4 shows summary of barriers and mechanism for increasing beneficial use of RMCs.

Applicable Types of **Barrier Category** Barrier Mechanisms **Economic Barriers** Transportation costs and i) Economic incentive geographic distribution ii) Procurement policies and RMC as minor component of building standards producers overall revenue iii) Education, technical Poor cost-effectiveness of RMC assistance, and utilization recognition Low cost of RMC disposal Legal, Regulatory, Federal air pollution regulations i) Procurement policies and State and Federal solid waste and Contractual building standards Barriers regulations ii) Education, technical Bidding procedures and assistance, and contractual constraints recognition Technical Barriers Performance of products i) Education, technical containing RMCs assistance, and Acceptance of materials recognition specifications ii) Economic incentives Variation in the quality of RMC supplies Health and Safety Perceived risk associated with i) Education, technical **Risk Perception** products containing RMCs assistance, and Barriers recognition

Table 3.4 Summary of barriers and mechanisms for increasing beneficial use of RMCs (EPA Report 2008)

Some of the analysis in the EPA report for the year 2004 and 2005 indicated reduced energy use of 31.5 billion mega joules, avoided CO_2 equivalent air emissions of 3.8 million metric tons, and water savings of 2.1 billion litres.

The Recycled Materials Resource Centre (RMRC) in the United States (US) has provided a list of specifications for use of industrial materials in highway applications. Some of these materials include: coal fly ash; coal bottom ash / boiler slag; foundry sand; ground granulated blast furnace slag (GGBSS) and reclaimed concrete. The coal fly ash, bottom ash / boiler slag or combinations of these industrial wastes are produced by coal-fired electrical or steam generating stations. The Indiana Department of Transportation (InDOT) Standard Specification includes the use of fly ash and GGBSS. Fly ash can be used as a pozzolan and its acceptance criteria is based upon the supplier's documented ability to consistently furnish material in accordance with American Association of State Highway and Transportation Officials (AASHTO) M295 for class C and class F. GGBSS can also be used as a pozzolan. Its acceptance criteria are based on the manufacturer's or manufacturer / distributor's documented ability to consistently furnish these materials in accordance with the applicable American Society for Testing and Materials (ASTM) and AASHTO requirements. Regards to the ASTM requirements, the slag shall be in accordance with C989 for grade 100 or 120.

Technical Advisory T5040.37, detailed information on guidance for the use of recycled concrete pavement as aggregate in hydraulic-cement concrete pavement. The terminology "hydraulic-cement" as opposed to "hydraulic bound mixture" as referred to in the UK are the same. Due to the use of blended cements, supplementary pozzolans and cementitious materials as binders, the term "hydraulic-cement" is used in the US.

(Saride, Puppala and Williammee 2010) reported that the total recycling process stream in the United States is estimated to be around 800 Mt per annum. They investigated the use of cement-stabilised quarry fines (CQF) and reclaimed asphalt pavement (RAP) as pavement base materials for a highway extension project in Arlington by the Texas Department of Transportation (TxDOT), USA. Laboratory and field studies were undertaken to evaluate their potential use as pavement bases. The laboratory investigations included compressive strength tests, swell / shrink and resilient modulus tests.

Based on the data collected in the laboratory investigations, both the materials were characterised as having very low swelling and compressibility values. The CQF exhibited a very low unconfined compressive strength (UCS) of 120 kPa. The strength improved 12-fold when treated with 2.3% cement additive. The RAP was observed to have UCS of 340 kPa. The resilient moduli values of CQF and RAP were between 300 and 400 MPa. Data received from the field tests revealed that the pressure of around 20 kPa recorded agrees with the expected values under traffic loading on pavement structures. It was concluded

that the results obtained for both the laboratory and field tests performed well to support heavy traffic load on rigid pavements.

3.4 Environmental Effects in using By-product in Concrete Pavement

3.4.1 Environmental Impact in using By-product

Industrial by-products may contain some form of varying proportions of chemicals. Some of these chemicals include organics and ferrous metals. There is a likelihood that these chemicals may leach and migrate to the surface and existing ground water which may affect their suitability for use in highway construction.

According to (Baldwin et al. 1997), "it has been recognised that many of these reclaimed materials and industrial by-products 'cause no significant environmental problems. However, materials containing water soluble salts may give rise to leachates at concentrations that would in some circumstances give cause for concern' (Williams, 1995)."

These by-products however, if used appropriately for instance in unbound pavement layer such as the foundation or in earthworks and far away from near-by water courses will act as mitigation measures. If these materials are used within bound applications such as hydraulically bound pavements, concrete and concrete blocks, this would reduce potential risks of leachates.

3.5 Base Materials Available in the Market

3.5.1 By-pass Dust

(Ganjian, Eshmaiel, Jalull and Sadeghi-Pouya 2015) defined by-pass dust (BPD) as a byproduct collected from the kiln bypass in the manufacturing process of cement. (Al-Jabri et al. 2006) defined BPD as "a by-product of the manufacture of portland cement. It is generated during the calcining process in the kiln. As the raw materials are heated in the kiln, dust particles are produced and then carried out with the exhaust gases at the upper end of the kiln." The chemical characteristics of BPD vary from different cement plant. This is attributed to the feed stock employed at the cements regard to the raw materials, kiln design, fuel type and the operation system used.

(Pouya et al. 2007) investigated a novel composite binder without using portland cement for binary and ternary groups of by-pass dust (BPD), plasterboard gypsum waste (PG) and basic oxygen slag waste (BOS).

In the binary group using a range of BPD and BOS with the same water content, they observed that mixture with 40% of BPD and 60% of BOS showed the highest compressive strength at 7 days. It was observed that an increase of BPD content upto 40% leads to an increase of the strength from 3.58 to 9.5MPa after 7 days of curing. The experimental results also revealed that the highest compressive strength at 28 days was still achieved by the mixture of BPD40 / BOS60.

For the ternary group of PG - BPD - BOS and BPD - PG - BOS, the highest compressive strength corresponds respectively to mixtures of (10% of PG, 36% of BPD and 54% of BOS) and (5% of BPD, 19% of PG and 76% of BOS).

3.5.2 Steel Slag Dust

Steel slag dust (SSD) is part of the ferrous slags family. Ferrous slags can be divided into blast furnace slags and steel slags. Blast furnace slag (BFS) is a by-product that arises in the production of iron. Steel slag is a by-product arising in the production of steel. Steel slag can be further sub-divided into basic oxygen steel (BOS) and electric arc furnace (EAF).

(Shi, C. 2004) describes the production and treatment process of steel slag as the byproduct from either the conversion of iron to steel in a basic oxygen furnace (BOF), or from the melting of scrap to produce steel in an electric arc furnace (EAF). Hot liquid metal from the blast furnace, scrap, and fluxes consisting of calcium oxide (CaO) and dolomitic lime are charged to a furnace in the BOF process. The impurities which consist of carbon monoxide, silicon, manganese, phosphorus and iron as liquid oxides combine with lime and dolomitic lime and are separated by injecting high-pressure oxygen to form the steel slag. In the EAF process, unlike the BOS process cold steel scrap is melted through electric current passing three graphite electrodes to form the arc.

(Motz and Geiseler 2001), the suitability of steel slags is stated by fulfilling the requirements of natural and / or international standards and regulations. In Europe alone, around 12 million tons of steel slag are generated every year. In Germany in 1998 about 97% of the produced steel slags have been used as aggregates for road construction, earthworks and armourstone for hydraulic structures.

(Ganjian, Essie et al. 2008) researched the optimum combination for a novel binder using synthetic sources as a by-product of industrial processes for construction of road foundations. The optimum combination for the novel binder achieved a mix of 15% recycled gypsum from waste plasterboard, 5% by-pass dust and 80% of basic oxygen slag. Using the novel binder, a semi-dry mix with a strength of 31MPa was achieved at 28 days. The novel binder was used as 100% cement replacement for road construction in two sites.

3.5.3 Gypsum

By-product gypsum reported in the literature are:

- i. Plasterboard Gypsum;
- ii. Flue Gas Desulphurisation;
- iii. Red Gypsum; and
- iv. Phosphogypsum

Out of the four by-product gypsum listed above, only two were used in this research: Plasterboard Gypsum and Flue Gas Desulphurisation (FGD). However, the remaining two were discussed briefly in this research.

3.5.3.1 Plasterboard Gypsum

WRAP report on plasterboard and gypsum waste states, "despite recycling of plasterboard waste (gypsum) being technically and commercially viable, approximately three million tonnes are landfilled in Europe annually."

(Ganjian, Essie et al. 2008) researched on the use of plasterboard gypsum waste in road bases. They described plasterboard as being made of a gypsum plaster core with a paper facing. Its application includes forming partitions, lining walls and ceilings. Over 2.5 million tonnes of plasterboard are manufactured and used in the UK each year, and this is a growing trend. Plasterboard waste arises during installation through wasteful design, off-cuts, damaged boards and over ordering. They further explained the wastage that usually occurs on site to be in the order of 10% - 35% and this leads to approximately 300,000 tonnes of plasterboard waste being produced each year from this source. It is estimated that in total more than one million tonnes of plasterboard waste is produced in the UK each year.

Emery et al. (2007) discussed the '10% rule' in their paper (demonstrating how plasterboard can be collected more efficiently on construction and demolition sites in the UK) and the effect the legislation has had since it came to force in July 2005. Plasterboard waste was included in a category of high-sulphate bearing wastes in the Landfill (England and Wales) Regulations 2002 (as amended). Mixed waste with a content of more than 10% sulphate per load may only be disposed of in high-sulphate 'mono-cells' separated from biodegradable waste in non-hazardous waste landfills. Some commentators saw this as a legislative barrier in the recycling of plasterboard waste and believe that the '10% rule' may have acted as a disincentive to the segregation of plasterboard waste from other construction and demolition waste.

The Coventry Walsgrave Hospital (Skanska Integrated Projects) was one of the three live projects mentioned in Emery et al's paper. The project involved the construction of an acute hospital for the University Hospital and Coventry and Warwickshire NHS Trust and a clinical sciences tutorial and research building. Two of the objectives of the study were to review the operational procedures for the recovery of plasterboard waste on-site and its economic benefits if any. The procedures for the management of plasterboard and plasterboard waste at this site are summarised in Figure 3.2.



Figure 3.2 Coventry hospital plasterboard material flow diagram (Emery et al., 2007)

Even though it was not possible for them to collect data on plasterboard use and accurately monitor the level of plasterboard wastage for the entire project, it was concluded however that between the period of January and October 2005, 1,365 tonnes of plasterboard were delivered to the site and 561 tonnes of plasterboard waste was generated in the same period. This equates to approximately 40% wastage rate over this period which is slightly above that mentioned in Ganjian et al. (2009) study for general wastage that usually occurs on construction sites in the UK. The average cost of transporting and transferring plasterboard for processing was estimated at £58/tonne. This

is less than the average £61/tonne cost of disposal of mixed waste to landfill encountered at the site.

Claisse et al. (2007) studied six site trials of sustainable concrete mixtures, two of these were for the applications in roads and car parks. One of the objectives of the study was to demonstrate how the sustainable concrete mixtures performed under site conditions.

The site trial that involved the access road was the stabilisation of soil with a sustainable "cement" as sub-base and then placing of a semi-dry paste (grout) as a road base with binder mixtures of plasterboard gypsum (PG), by-pass dust (BPD) and basic oxygen slag (BOS). In order to monitor its performance, half of the 22m length of the site access road was constructed in conventional base material with the same thickness as the semi-dry compacted paste. The mix proportions for the soil stabilisation and semi-dry paste were (soil 50% / binder 50%) and (PG 15% / BPD 5%/ BOS 80%) respectively. Mobile sprinkler was used in order to obtain the optimum compaction of the stabilised soil. For the base mixture, water content of the paste was limited to 13%.

Laboratory tests for the stabilised soil was carried out at 3, 7 and 28 days. There was insignificant difference in the early strength of the stabilised soil compared to the unstabilised soil. The long-term strength however, was very significant. The highest strength of 5.98 MPa compared to 0.13MPa at 28 days was achieved.

Laboratory and in-situ tests were carried out for the semi-dry paste. Tests were carried out at 3, 7 and 28 days for the lab tests and 14, 28 and 90 days for the in-situ tests. Both the laboratory and in-situ tests showed that the strength of the material increases in time. The highest compressive strength obtained in the laboratory test was 30.55MPa and that obtained in the in-situ was 46.41MPa.

Sadeghi Pouya et al. (2007) carried out a study that explored the potential benefits of utilising combinations of plasterboard waste gypsum and pozzolanic materials as novel cementitious pastes. The aim of the study was to develop novel cementitious binders using plasterboard gypsum waste (PG), basic oxygen slag (BOS) and cement kiln dust (CKD) / by-pass dust (BPD) for road base applications. For the ternary groups of PG-BPD-BOS and PG-CKD-BOS, the highest compressive strengths correspond respectively to

mixtures of (10% of PG, 36% of BPD and 54% of BOS) and (16% of PG, 20% of CKD and 64% of BOS). It was observed that increasing the amount of PG above 10% in the PG-BPD-BOS mixture resulted in decrease in compressive strength. It can also be seen from the test results for the PG-CKD-BOS mixture that as a result of increasing the amount of CKD from 40 to 60%, compressive strength has been reduced by about 50% at 28 days. The highest compressive strength obtained from the mixtures of PG10/BPD36/BOS54 and PG16/CKD20/BOS64 were 8MPa and 13MPa respectively.

(Ganjian, Essie et al. 2008) carried out a research study as part of Waste Resources Action Programme (WRAP) Plasterboard Programme to divert plasterboard waste from landfill, to be used in a number of road construction applications. Laboratory testing and a series of on-site trials were carried out on a number of sites in the West Midlands. In this study, the optimisation for proportions of recycled gypsum and other mineral wastes were used to achieve the highest compressive strength. According to their investigation, the optimum combination of these materials was a mix of 15% plasterboard derived from recycled gypsum (PG), 5% BPD and 80% BOS to achieve a novel binder. Using the novel binder with 13% water, the highest compressive strength of 31MPa at 28 days for the base in the road site trial was achieved.

(Pouya et al. 2007) carried out a similar study to that of Karami et al. (2007) using plasterboard gypsum waste (PG), cement by-pass dust (BPD) and basic oxygen slag waste (BOS). The waste materials were analysed in terms of strength development to establish the optimum binary and ternary mixture proportions corresponding to the highest compressive strength. One of the applications for the low strength cementitious material was for it to be used in pavement bases. It was observed that in optimising BPD-PG-BOS group, the highest compressive strength obtained was for the mixture of 5% BPD, 19% PG and 76% BOS, the same optimal percentage as that achieved in Karami et al. (2007) study in Section 3.5.3.3. The study showed that the increase of BPD content has no significant effect on compressive strength at early ages (3 and 7 days), however, at 28 days, mixtures with less BPD content showed greater compressive strength.

3.5.3.2 Flue Gas Desulphurisation

(Plunk 2016) defined Flue Gas Desulphurisation (FGD) as a residue remaining after flue gas undergoes sulphur removal (desulphurisation). At coal-fired electrical generating stations, this desulphurisation process is designed to remove sulphur dioxide SO_2 from exhaust flue gases by injecting $CaCO_3$ (limestone) into the hot flue gases. The calcium carbonate reacts with the sulphur in the flue gases, producing $CaSO_3$ (calcium sulphite). The calcium sulphite can be further oxidised to yield calcium sulphate dehydrate (CaSO₄. 2H₂O) by using forced oxidation. The calcium sulphate dehydrate is more commonly referred to as synthetic gypsum.

Vandivort et al. (2007) "more than 123 million tonnes of solid by-product are produced by coal-burning electric utilities each year in the United States. In particular, the annual production of flue gas desulphurisation (FGD) by-products continues to increase. As landfill space becomes more limited and expensive, there is incentive to find productive and sustainable uses for coal combustion by-products (CCBs)."

They defined flue gas desulphurisation (FGD) as a solid residue material which is produced when sulphur dioxide gas is removed from power plant boiler stack gases during the flue gas desulphurisation process. The FGD material develops in flue gas scrubbers when gaseous sulphur dioxide reacts with slurried limestone to produce calcium sulphite (gypsite). Further oxidisation of the calcium sulphite becomes calcium sulphate, or gypsum.

The British Geological Survey Mineral Planning Factsheet on gypsum notes that "synthetic gypsum (calcium sulphate) may also be derived as a by-product of certain industrial processes. The most important is flue gas desulphurisation (FGD)." As referred to as on (BGS online), the product, desulphogypsum, is an important supplement to the supply of natural gypsum, both in the UK and elsewhere. It was noted that synthetic gypsum has a higher purity (gypsum content of 96%) than most natural gypsum of 80%) in England.

In the U.S. in a 2014 survey, FGD gypsum production increased from 9.7 million to 34.1 million tons as emission-control devices were added and operated at coal-fired power

stations. FGD gypsum use increased in 2014 from 4.8 million to 16.8 million tons (Plunk 2016).

In the UK, the output of desulphogypsum was a record 1,228,000 tonnes in 2004 and will increase further as new FGD plants come to stream.

In general, the production of FGD depends highly on the electricity station or other industrial processes output and the sulphur content of the coal. The quality and purity however, of the residue produced varied widely depending on fuel composition, process design and collection method. Depending on the configuration, the consistency may vary from dry powdered material to wet sludge. Some of the environmental benefits in the use of by-product gypsum can be summarised as follows:

- remove poisonous emissions from the air that has been generated through electricity stations and other industrial processes;
- reduce haulage costs and improve the environment in seeking naturally occurred or mined gypsum; and
- reduce the amounts of industrial by-products that are being sent to landfill sites not only in the UK but elsewhere.

(Tokalic et al. 2012) carried out an investigation into the use of three power plant wastes: flue gas desulphurisation (FGD); fly ash (FA); and bottom ash (BA). Their study found potential use of these materials for sub-base layers in road construction. The paste mixtures fly ash-Portland cement-flue gas desulphurisation gypsum-bottom ash-water (FA-PC-FGD gypsum-BA-W) were made using (1) raw fly ash (consisting of particles smaller than 1.651mm) and (2) sieved fly ash (consisting of particles smaller than 0.42mm). Both mixtures had the same mass composition (FA:PC:FGD gypsum:BA = 3:1:1:5). The mass composition was 18.6%FA- 6.2%PC- 6.2%FGD gypsum- 31%BA-38% W.

The compressive strength for both mixtures were determined 7 and 28 days after preparation. The investigation revealed that the specimen made with FA consisting of

smaller particles had greater compressive strength values than the specimen consisting of bigger particles. It was evident that the compressive strengths of both samples after 28 days (3.1MPa) of hardening were higher than those after 7 days (1.5MPa) by almost 200%. The higher compressive strength the researchers believed, was probably the higher reactivity of the smaller particles and consequently higher content of the products of hydration reactions. It was concluded that both mixtures were suitable and satisfied the criteria for sub-base layers for medium and heavy traffic load.

(Tao and Zhang 2005) evaluated the mechanical properties of stabilised blended calcium sulphate (BCS), a by-product gypsum, blended with lime or limestone. This blended by-product was used as road (base) material. BCS was satisfactorily stabilised with various cementitious materials, such as ground granulated blast furnace slag (GGBSS), Portland cement and fly ash. Durability tests demonstrated that BCS stabilised with 10% GGBSS can sustain long-term satisfactory performance after it has cured for 28 days.

3.5.3.3 Red Gypsum

Karami et al. (2007), "Titanium Oxide Pigment Production yields 250kt of 'red' and 84kt of clean 'white' gypsum per year in the UK. Worldwide production of red gypsum is 1.25Mt from one producer alone. This material contains approximately 40% moisture, 16% iron oxide, 0.5% of both $Mn0_and SiO_2$, 0.25% of Al and TiO_2 and many other elements. This renders it unacceptable to the plaster and cement industries, principally due to its iron content which may cause staining in plaster products and adversely affects cement clinker chemistry."

British Geological Survey, referred to red gypsum as titanogypsum, a by-product of the manufacture of titanium dioxide pigment and is another source of synthetic gypsum. This by-product is produced by neutralising acid effluent with chalk arising from the manufacture of titanium dioxide pigment by the Sulphate Process. Even though this waste material has a very high iron content which may cause staining in plaster products, BGS (online) reported that a plant in Grimsby (UK) produced approximately 100,000 t/y of high purity gypsum and used at Immingham (UK) for plasterboard manufacture.

A study carried out by Karami et al. (2007) investigated the use of industrial waste materials such as Plasterboard Gypsum (PG), Red Gypsum (RG), Basic Oxygen Slag (BOS) and Cement By-pass Dust (BPD) in construction. One of the objectives of the study was to develop uses of waste industrial materials. In order to find the optimal percentage of each material, the materials were mixed in binary and ternary combinations and the tensile strength and compressive strength was tested and optimised.

In optimising the ternary mixes, each mix was a combination of three different materials. The combinations were BOS–BPD–RG and BOS–BPD–PG. Compressive strength of the samples were tested at 3, 7 and 28 days. In all the mixes it was evident that the compressive strength has increased by curing time. In other words, 3 days old samples have lower compressive strength than 7 days old samples and 7 days old samples have lower compressive strength than 28 days old samples.

In the group of BOS–BPD–RG, it can be observed that the mixture achieved a long-term strength of 2.70MPa at 28 days of curing. The optimal percentage of this was BOS(85)– BPD(5)–RG(10). On the other hand, the Group of BOS – BPD – PG achieved a much higher compressive strength of 4.5MPa at 28 days of curing. The optimal percentage however, was BOS(76)–BPD(5)–PG(19). It was observed in the study that mixes that contained PG can achieve higher compressive strength after 28 days compared to the same mixes which have RG.

(Claisse, P., Ganjian and Tyrer 2008) examined the strength properties of controlled low strength materials (CLSM). The principal waste material used in the research was "Red" gypsum. Other waste materials were discussed for mixing with the red gypsum to make it set. Initial laboratory mixing trials were carried out with numerous cup mixes. The most promising and successful waste was Basic Oxygen Slag (BOS) from steel production.

CLSM is generally used for backfilling in trenches, under pavements and around pipes which usually requires an unconfined compressive strength of 2.1MPa or less. The highest compressive strength of 2.8MPa for one of the candidate mixes for the trench fill trial site was obtained at 90 days. The mixtures were 30% of RG, 60% of BOS and 10% of CKD.

Electron Microscopic and X-Ray Diffraction Experiments were carried out on selected samples. Analysis of the experiment shows that samples with high water to solids ratio created sufficiently high porosity to prevent a continuous bond between the gypsum binder and the steel slag aggregate and were therefore disregarded. The other formulation used in the site trial whose microstructures show no features, which may raise concern about the material's future performance was a binary mixture with 40% of RG and 60% of BOS.

3.5.3.4 Phosphogypsum

(Huang and Lin 2010) defines phosphogypsum as "a by-product generated by phosphorus fertilizer industry. It consists mainly of $CaSO_4$. $2H_2O$ and contains some impurities such as P_2O_5 , F⁻, and organic substances. The quantity of phosphogypsum is large: about 5 tons of phosphogypsum is produced for each ton phosphate acid."

They used phosphogypsum (PPG) in combination with ground granulated blast furnace slag (GGBSS), steel slag (SS) and limestone (LS) to investigate low energy consumption cement. The research was to investigate the soundness and compressive strength together with the setting time and water demand of the cement. The microstructure and hydration of the cement was studied using XRD and SEM.

It was concluded in the study that the highest compressive strength achieved at 28 days was for mixture of 45% PPG, 10% SS, 35% GGBSS and 10% LS. The strength obtained exceeded 40MPa. The XRD patterns studied for the specimen with the highest compressive strength at 28 days revealed that the main crystalline components in the hydrated paste were ettringite and residual PPG. The SEM images however, show that a large amount of needle-shape ettringite was formed on PPG surface at early ages. This connected the PPG particles and hydration product and increase the interface binding strength.

3.5.4 Reclaimed Asphalt Fillers / Aggregates

While the use of reclaimed asphalt pavement (RAP) as aggregates replacement in concrete have been widely studied in labs: (Arshad and Ahmed 2017), (Pradyumna, Mittal and Jain 2013), (Chandra, Veeraragavan and Krishnan 2013) and (Hajj, Sebaaly and Kandiah 2009), research into the use of reclaimed asphalt fillers in hydraulically bound
pavements are considered fairly new. Therefore, the need to fill the knowledge gap of research in this area is of immense interest to not only researchers in the academic forum, but also the construction and other manufacturing industries. The wider use of the fillers with potential as cement binders would contribute heavily to address sustainable solutions in the efficient recovery of road layers in bituminous pavements.

(DMRB HD35/04 2004), states that reclaimed bituminous materials can be used for hot mix recycling in the production of bituminous base, binder and surface course and up to 10% of reclaimed bituminous materials being permitted without the requirement for trials. Recent research has shown that a high percentage of RAP might be detrimental to the overall performance of the pavement. According to (Howard, Doyle and Cox 2013), approximately 85% of the RAP available is used within either hot mix asphalt (HMA) or warm mix asphalt (WMA).

Reclaimed asphalt is derived from planings of worn-out flexible surface course (approximately 40 – 50mm deep) or milling / excavation of the existing flexible pavement to a much greater depth if the pavement shows evidence of structural distress such as rutting, shoving, depressions, potholes, longitudinal and transverse cracks etc. Reclaimed asphalt filler (RAF) is a material that is produced by the manufacturers' Via Nova Asphalt Plant and conveyed to the plant reclaimed duct silo. The material is a natural calcined pozzolan and is formed during the processing of asphalt using sedimentary rocks. The material is classed as filler when approximately 80% passes through a 0.063 mm sieve. Some of these materials may be used as one of the recipe ingredients to produce asphalt.

One of the objectives of the research project carried out by (Aggregate 2013) for the Illinois Department for Transport was to determine the expansive properties of RAP materials, particularly those with recycled steel slag aggregates that could be used as pavement base materials and the effects that RAP materials may have on pavement performance. The laboratory studies indicated that steel slag aggregates showed a potential for expansion of up to 6.2%, which is higher compared to virgin aggregates. Conclusions from the expansion test revealed that RAP materials with slag aggregates that was partly coated with asphalt had much lower tendency to expand than the virgin steel slag aggregates, which showed high expansion potentials.

(Shi, X., Mukhopadhyay and Zollinger 2018) carried out sustainability assessments of the use of reclaimed asphalt pavement in Portland cement concrete (PCC) as an aggregate replacement for pavement application. The assessment was carried out through the economic input out - life cycle assessment (EIO-LCA) approach. Results from the research demonstrated that one of the three pavements used could yield the highest economic benefits. While the other two pavements could have the highest positive impacts from social and environmental perspective. One of the conclusions from the study revealed that in using conventional pavement design tools, the pavement that could yield the highest economic benefits had a thicker slab compared to a plain PCC pavement. The study showed that in using RAP-PCC as the bottom lift material in a two-lift concrete pavement can maximise the use of RAP with minimum compromise to the performance of the pavement.

(Arulrajah, Piratheepan and Disfani 2013) researched the use to incorporate RAP and recycled concrete aggregates (RCA) into pavement base and subbase by means of cement binder stabilisation. They investigated RAP with contents of 100, 50, 30 and 15% in blends with RCA. Results of the laboratory evaluations of the geotechnical properties of RAP-100 indicated that it had high water absorptions values compared to RCA–100. RAP-100 was also observed to have a relatively high loss on ignition, this may have been attributed to the presence of bitumen. The study also reported on the dry density-moisture content relationships. It was found that RAP-50/RCA-50 had a maximum dry density (MDD) of 19.9 KN/m3 which was the highest. The lowest MDD of 19.03 KN/m3 was for the blends RAP-30/RCA-70. It was found that the hydraulic conductivity values did not vary with the percentage of RAP of RCA contents. The highest and lowest hydraulic conductivity values of 7.45 X 10^{-7} and 3.3 X 10^{-8} m/s were obtained for RAP-50/RCA-50 and RCA-100 respectively.

3.5.5 Quarry Waste Dust

(DMRB HD35/04 2004), defines quarry fines as being derived from processing of crushed rock for use as construction aggregate consists of blasting, crushing, washing, screening and stockpiling operations.

(Shyam Prakash and Rao 2016) defines quarry dust (QD) as a by-product derived from the crushing process of rock into numerous sizes. During this crushing process, the dust

generated is formed as quarry waste dust (QWD). Their study on QWD investigated the influence of different replacement proportion of sand with QD on properties of concrete. In the study, the compressive strength was quantified for varying percentage and grades of concrete by replacement of sand with QD.

A set of five series of compressive tests were carried out to achieve the following grades of concrete: M20; M25; M30; and M40. The percentage replacement of QD were 20, 25, 30 and 40 in the first four sets and 10 percentage increment up to 100 percentage replacement in the fifth set. The samples were tested for 3 days, 7 days, 28 days and 60 days. Water cement ratio (w/c) used was 0.45 and 0.50.

Results from the experimental study concluded that cement with a w/c ratio of 0.45 has higher compressive strength compared with w/c of 0.50. This was due to the water absorption property of QD. Regarding the percentage of sand replacement, it was observed in the study that 40% replacement gives a maximum result in strength compared to normal concrete and then decreased from 50%.

(Zou, Xu and Wu 2013) carried out a research to evaluate the feasibility of using quarry waste (QW) for pavement applications. They described QWs used in their study as "mainly composed of chippings, top layer soil, clay and some coarse particles." The source of this waste is different from that studied by (Shyam Prakash and Rao 2016); a quarry waste dust (QWD) which was derived through the rock crushing processes of different sizes of aggregates used in concrete and flexible pavements.

The results from their investigatory study revealed that QWs cannot be used as subbase because it did not meet the required specifications. However, it was noted that the QW materials can be used for subgrade improvements. The field testing carried out showed the resilient modulus of the 800mm QW improved subgrade 70% higher than the original subgrade. Observations of the results from the unconfined compressive strength at 7 days on core samples was an average 3.14MPa, this met the required strength specification.

3.5.6 Limestone

(Taylor 2002), defined limestone as predominantly calcium carbonate and are formed when calcareous deposits are cemented together underwater by further calcium dissolved in the water.

Limestones are non-toxic minerals and white in colour. They can be easily ground to a fine powder. They are sedimentary rocks in a form of the mineral calcite.

3.5.6.1 Production of Limestone in Great Britain

In Great Britain, limestone (including dolomite) accounts for almost half of crushed rock aggregate produced.

BGS online on limestone states that "detailed production data for limestone and chalk for industrial and agricultural purposes by individual use are not publicly available because of confidentiality considerations. However, in 2004 it is estimated that a total of 9 million tonnes were produced in Great Britain, of which 77% was limestone and 23% chalk. For industrial use limestone accounted for 72% (6 million tonnes) of the total."

3.5.6.2 Uses of Limestone

One of the largest uses of limestone is in the iron and steel industry. It is used as flux in iron and steel making. Due to the decline in steel production in Great Britain, the consumption of limestone in this sector has decreased.

Limestone is mainly used in the construction industry as crushed aggregates for road base. They can be used as one of the raw materials for cement manufacture. They can also be used as railroad ballast.

When ground into fine powder, it can be highly cementitious and can be used as a filler in both concrete and asphalt applications or as an activator to pozzolanic materials. Some of its highway applications include:

- asphalt concrete mineral filler;
- asphalt cement modifier;

- stabilized earthworks; and
- flowable fill

Below are some other uses for filler applications other than highway:

- acting as a source of magnesium in pharmaceutical products such as magnesium salt;
- acting as a source of calcium in animal feeds;
- acting as an acidity regulator (pH control) in agriculture;
- manufacturing of carpet backing, floor tiles, sealants, adhesives, putties, paper fillers, paper coatings, paints, rubber and plastics.

BGS online sates that "a particular important market for high purity limestone, which has emerged in the last 10 years, is flu-gas desulphurisation. This involves the removal of sulphur dioxide contained in the flue gases at coal fired power stations, in a water based slurry of finely ground limestone. Gypsum is produced as a by-product".

3.5.6.3 Limestone Chemical Formula – Thermal Decomposition and Hydration

3.5.6.3.1 Thermal Decomposition of Limestone

Calcium carbonate breaks down when heated to high temperature in specially designed kilns. Through the burning process and at temperature of 900 degrees Celsius, carbon dioxide gas is released leaving behind calcium oxide. The calcium oxide is also referred to as 'quicklime' or pulverised lime. Below are the chemical equations for the thermal decomposition of calcium carbonate:



Figure 3.3: Chemical reaction of calcium carbonate when heated

Equations for reaction in Figure 3.3:

$$CaCO_3 \xrightarrow{Heated} CO_2 + CaO$$
 (Equation 3.1)

3.5.6.3.2 Hydration of Quicklime

The pure oxide form, called quicklime is produced by heating pure limestone to a high temperature. When the quicklime is hydrated, its reaction with water forms calcium hydroxide. The hydrated quicklime is also referred to as slaked lime (terminology derived through the process of 'slaking' with water). Below are the chemical equations of the hydration process:



Figure 3.4: Chemical reaction of quicklime and water

Equations for reaction in Figure 3.4:



3.5.6.3.3 Chemical Composition of Dolomite

Limestone grades into dolomite with an increase in magnesium carbonate. The chemical reaction of calcium carbonate and magnesium carbonate give calcium magnesium carbonate. Below is the resultant of the dolomite mineral:



Figure 3.5: Chemical reaction of calcium carbonate and magnesium carbonate

Equations for reaction in Figure 3.5:

$$CaCO_3 + MgCO_3 \longrightarrow CaMg(CO_3)_2$$
 (Equation 3.3)

3.5.6.4 Lime Kiln Dust and Hydrated Lime

Limestone (calcium carbonate) is mainly used in the construction industry as crushed aggregates in road base and earthworks. When burned in lime kiln, the calcium carbonate breaks down. The results of the thermal decomposition process is explained in Section

3.5.6.3.1.The calcium oxide that is formed through the burning process is also referred to as quicklime (QL). Lime kiln dust (LKD) is a by-product produced through the manufacturing of QL. LKD with high free lime content is reactive when water is added, this can sometimes act as a substitute for hydrated lime.



Figure 3.6: Lime Hydrating Plant (Tarmac 2016)

When water is added to the quicklime, the process is known as slaking. When the quicklime is hydrated, its reaction with water forms calcium hydroxide also referred to as slaked lime. *Figure 3.6* shows a schematic diagram of a lime hydrating plant.

(Latif et al. 2015) investigated the effect on strength in replacing ordinary Portland cement

(OPC) up to 60% by weight with lime kiln dust (LKD). The study revealed that at 28 days, LKD obtained strengths above 50MPa. This was achieved in replacing cement with up to 50% LKD. Reduction in strength however, was attributed to the cement being replaced by LKD to a much higher percentage. The study concluded that higher amount of LKD affects the overall strength performance. The conclusion also included that adding LKD as a cement replacement accelerated the initial and final setting times significantly.

(Abdul and Timothy 2005) investigated the use of LKD for stabilisation of subgrade soils. One of the objectives of the investigation was to determine the engineering properties of LKD – soil mixtures, containing 2% to 10% by weight. Another objective was to investigate the effect the long-term ambient curing has on the unconfined compressive strength of LKD-soil mixtures.

In order to determine the optimum LKD content for the three different types of LKD materials obtained from three different sources, each LKD was mixed with the soil at 2% intervals. Results from the study revealed that the strength values for all the LKD-soil mixtures follow very similar trends. It was observed that the unconfined compressive strength values increase with increasing LKD content and gradually level off at 6% LKD content. The strength values for the three different LKD-soil mixtures at LKD content of 6% and above were in the range of 620 to 689 kPa for the first two mixtures and 551 kPa for the third when compared to the strength of pure soil which was 381.2 kPa.

Using the 6% dose of each of the LKD materials, the mixtures were compacted and allowed to cure for 3, 7, 9, 27 and 81 days at ambient conditions. Results of the time-dependent unconfined compressive tests showed that the strength increases with time. The early strengths obtained at 3-day curing period was in the range of 552 to 621 kPa. All the samples had unconfined compressive strengths of more than 827 kPa after a 9-day curing period. It was concluded in the study that for modification of subgrade soil, a 4% by weight dosage of LKD may be adequate where as 6% LKD content is required for more complete stabilisation.

3.5.7 Ground Granulated Blast Furnace Slag (GGBS)

GGBS is a well-known constituent of concrete, frequently used to enhance the properties of concrete as well as substituting for Portland cement. It can be used with lime as a cementitious combination for the treatment of granular materials (Kennedy John 2006).

GGBS is a waste product in the manufacture of pig iron, about 300 kg of slag being produced for each tonne of pig iron. Chemically, slag is a mixture of lime, silica and alumina, that is, the same oxides that make up Portland cement but not in the same proportions (Neville 2011).

(Singh, Tripathy and Ranjith 2008) investigated cement stabilised fly ash – granulated blast furnace slag (GBFS) mixes for earthworks, sub-base and base materials for highway construction. The cement content was varied from 0% to 8% at 2% intervals and the slag content varied at 0% to 40% at 10% intervals. A California Bearing Ratio (CBR) value of 105% was obtained for a mix of fly ash / GBFS / cement in the ratio of 52 / 40 / 8. It was observed there was a gain in compressive strength with addition of GBFS at constant cement content. This was due to the reactive siliceous and aluminous materials in the GBFS.

3.6 Summary of Review

Some of the researchers achieved their objectives in using industrial by-products as a complete or partial replacement to cement or as an alternative to naturally occurring materials.

A review of the literature reveals that research has been carried out on the use of plasterboard gypsum, by-pass dust and steel slag dust as cement replacement materials. Some of the detailed research that has been carried out on these materials show their successful applications in pavement as road binder, road base, as sub-grade materials and in the manufacturing of block paving and slab. Both plasterboard gypsum and by-pass dust are alkaline, and because of their alkalinity, they react well with steel slag which has pozzolanic properties to form a cement hydration process.

Extensive studies have been carried out on the use of RAP as aggregate replacement in

flexible and concrete pavements.

Review of the literature also indicated that moderate research has been carried out on lime kiln dust and quarry waste dust. Both these materials have been used to either partially replace cement or used for stabilisation of subgrade soils as foundation layers.

All but four of the industrial by-product materials in this research have been used in other research studies: MHT; LHT; V-B5G and RAF. Limited research has been carried out in the UK on the use of hydrated lime and asphalt filler, hence there is a need to not only further the studies of synthetic gypsums, by-pass dust and steel slag dust, but to also investigate and evaluate these new industrial by-products for their suitability in civil engineering applications particularly in pavement foundation and road base as hydraulically bound binder.

CHAPTER 4 - LABORATORY EXPERIMENTS ON MATERIALS SUITABILITY

4.1 Introduction

The chemical composition and physical properties of the materials used in this research study is explained in this chapter. The chemical composition of similar materials used in different studies carried out by other researchers are detailed.

Different types of laboratory experiments used to determine the suitability of the industrial by-products obtained were discussed. Material assessments were carried out based on detailed chemical and mineralogical properties of the samples.

4.2 Testing Methodology for By-product Suitability

The methodology in Figure 4.1 identifies the suitability of the by-products obtained. Materials identified to have pozzolanic and / or cementitious properties were further explored in the research study. Materials that have neither pozzolanic nor cementitious properties can be used as coarse or fine aggregates if they are considered to have satisfactory mechanical properties that is resistant to crushing.



Figure 4.1: Methodology for identifying suitable by-product

4.3 Chemical Composition of Materials from Other Researchers

This section provides data from other researchers. The chemical oxides or chemical compositions of the materials discussed in this section are not used in this research study. The materials discussed in this section can not necessarily be compared to the materials used in the research study as variations could affect the results.

4.3.1 Chemical Properties of By-pass Dust

Below are chemical oxides composition used in studies carried out by other researchers.

Table 4.1: Chemical Oxides Composition (Limbachiya, Ganjian and Claisse 2015)

Composition (%)	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO₃
BPD - July	12.79	0.19	3.47	1.88	0.04	0.82	44.03	1.16	10.06	0.12	12.22
BPD - August	15.58	0.22	4.26	2.26	0.04	0.98	50.32	0.70	5.80	0.13	9.23
BPD - September	14.85	0.20	3.70	2.11	0.04	0.85	47.43	0.88	7.46	0.12	13.42
BPD - October	15.13	0.21	3.84	2.24	0.04	0.93	53.13	0.66	5.09	0.14	6.25
BPD - November	16.52	0.23	4.17	2.29	0.05	1.11	49.57	0.71	6.29	0.14	11.18
BPD - December	17.34	0.20	3.75	2.35	0.05	1.05	52.75	0.52	4.03	0.15	9.94
BPD - T	16.85	0.23	4.16	2.36	0.04	1.04	53.60	0.50	4.28	0.15	6.66

4.3.2 Chemical Properties of Steel Slag Dust

Below are chemical oxides composition used in studies carried out by other researchers.

Table 4.2: Chemical Oxides Composition (Limbachiya 2015)

Composition (%)	SiO₂	Al ₂ O ₃	Fe₂O₃	MgO	CaO	Na₂O	K₂O	SO₃
1	18.01	2.61	14.10	14.10	34.02	-	-	0.35
2	18.87	2.91	11.73	14.37	37.94	-	-	0.37
3	17.09	4.56	-	10.46	40.46	0.42	-	0.00
4	17.53	6.25	26.36	6.45	35.70	0.20	0.26	0.75
5	11.43	1.60	28.24	8.27	41.29	0.02	0.02	0.44

4.3.3 Chemical Properties of Plasterboard Gypsum

Below are chemical oxides composition used in studies carried out by other researchers.

Table 4.3: Chemical Oxides Composition (Karami et al. 2013)

Composition (%)	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO₃
	2.43	0.03	0.81	0.36	0.40	37.30	0.03	0.24	0.02	53.07

4.3.4 Chemical Properties of Ground Granulated Blast Furnace Slag

Below are chemical oxides composition used in studies carried out by other researchers.

Composition (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	SO₃
1	35.50	14.70	-	-	45.30	-	-	2.10
2	36.10	11.70	1.20	7.30	40.10	0.47	-	1.90
3	35.46	4.73	2.93	2.67	41.83	-	-	-
4	39.00	14.00	0.80	9.00	32.00	0.35	1.20	1.50
5	34.00	11.11	0.50	11.60	36.60	-	-	3.30

Table 4.4: Chemical Oxides Composition (Limbachiya 2015)

4.3.5 Chemical Properties of Lime Kiln Dust and Large Hydrated Tailing

Table 4.5: Chemical Oxides Composition LKD and LHT

Composition (%)	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO₃	LOI
LKD	0.91	0.01	0.22	0.25	0.36	56.74	<0.013	0.01	0.00	0.38	41.50
LHT	0.43	<0.007	<0.022	0.03	0.40	60.76	<0.013	<0.005	<0.001	0.51	38.65
1											

4.4 Chemical Composition of Other Materials used in this Research

The following materials were sent to Leicester University for XRD and XRF analysis in 2017: V-B5G; RAF and MHT (Medium Hydrated Tailing). Further tests were carried out by the University of Warwick (UoW) for XRD and XRF analysis on the following materials in 2018: PG; QWD; RAF; SSD; and V-B5G. Additional tests on the V-B5G was instigated by changes to the mixture of the manufacturer's upstream wastes before the neutralisation / precipitation and filtration process that is likely to have changed the chemical composition of the calcium sulphate. Additional tests on the RAF was instigated to determine the consistency of the material regards to its manufacturing process.

XRD test determines the phases or component combinations of the mineralogical properties of a given sample. XRF test on the other hand determines the percentage of chemical composition present in a sample.

4.4.1 Chemical Properties of BPD-C

4.4.1.1 X-Ray Fluorescence Analysis of BPD-C

Composition (%)	SiO₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na₂O	K ₂ O	SO₃	CI	LOI
13 Jan. 2016	13.58	4.23	2.61	0.96	47.13	0.30	3.13	11.61	2.25	6.60
20 Jan. 2016	10.98	3.41	1.83	0.90	30.98	1.45	7.72	11.01	9.99	6.68
25 Jan. 2016	10.78	3.16	1.67	0.86	26.24	1.97	9.16	10.88	12.61	4.62
03 Feb. 2016	13.43	3.82	2.21	0.98	43.83	0.71	5.26	5.71	5.79	7.73
11 Feb. 2016	10.22	3.20	1.67	0.86	31.14	1.41	7.73	13.24	9.04	4.52
18 Feb. 2016	10.96	3.12	1.66	0.84	25.12	2.13	8.58	13.07	12.05	4.79
22 Feb. 2016	12.27	3.55	1.99	0.92	34.20	1.27	6.65	11.92	7.96	5.13
29 Feb. 2016	14.64	3.85	2.41	0.94	43.59	0.83	4.50	8.95	4.85	5.02
07 Mar. 2016	13.29	3.57	2.17	0.94	42.65	0.79	5.04	10.44	5.06	4.64
15 Mar. 2016	13.91	3.86	2.20	0.93	39.56	1.12	5.49	9.11	6.45	5.38
21 Mar. 2016	13.85	3.80	2.22	0.97	43.35	0.85	5.55	4.88	6.20	5.76
31 Mar. 2016	13.64	3.72	2.24	0.97	39.08	0.81	5.45	11.29	5.89	4.17
05 Apr. 2016	11.57	3.22	1.74	0.92	35.04	1.27	7.33	7.38	8.79	6.42
11 Apr. 2016	14.67	3.75	2.40	1.02	46.31	0.68	4.51	5.48	4.65	4.64
21 Apr. 2016	14.12	3.92	2.26	1.00	47.71	0.51	4.33	6.81	4.15	4.96
26 Apr. 2016	13.19	3.62	2.16	0.95	40.73	0.76	5.34	8.69	5.71	4.01
03 May. 2016	12.82	3.82	2.16	1.00	48.79	0.51	4.28	5.58	4.35	5.52
09 May. 2016	10.44	3.20	1.79	0.92	38.35	0.92	6.33	9.60	7.07	6.08

4.4.2 Chemical Properties of SSD

4.4.2.1 X-Ray Diffraction Analysis of SSD

The mineralogical composition of SSD include larnite, srebrodolskite, aluminian, qingheiite, brownmillerite and ferrian. The peaks and valleys pattern of the SSD showed the presence of high levels of calcium silicate $Ca_2(SiO_4)$, a moderate amount of calcium aluminium iron oxide ($Ca_2Fe_{1.592}Al_{0.408}O_5$) and low levels of magnesium manganese oxide (MgMn₂O₄).



Figure 4.2: XRD of SSD

4.4.2.2 X-Ray Fluorescence Analysis of SSD

Table 4.7: XRF 01 55L

Composition (%)	SiO₂	TiO₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K₂O	P ₂ O ₅	SO₃	MnO
SSD	9.80	0.84	2.30	26.00	3.40	43.00	0.02	0.02	0.93	0.23	2.80

4.4.3 Chemical Properties of PG

4.4.3.1 X-Ray Diffraction Analysis of PG

The mineralogical composition of PG are gypsum, anhydrite and quartz. The peaks and valleys pattern of the PG showed the presence of high levels of calcium sulphate dihydrate (CaSO₄-2H₂O), moderate levels of anhyrous calcium sulfate Ca(SO₄), and low levels of amounts of basanite or hemihydrate (CaSO₄-0.5(H₂O)) and silicon oxide (SiO₂).



4.4.3.2 X-Ray Fluorescence Analysis of PG

Table 4.8: >	(RF o	f PG
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Composition (%)	SiO₂	TiO₂	Al ₂ O ₃	Fe₂O₃	MgO	CaO	Na₂O	K ₂ O	P ₂ O ₅	SO₃	LOI
PG	0.96	0.03	0.31	0.27	0.13	40.00	0.02	0.09	0.02	52.00	-

4.4.4 Chemical Properties of V-B5G

4.4.4.1 X-Ray Diffraction Analysis of V-B5G

The mineralogical composition of V-B5G before changes were made to the upstream wastes were basanite and gypsum only. After changes were made to the manufacturing process, composition are calcite, quartz and gypsum. The peaks and valleys pattern of the V-B5G as a result of the changes to the manufacturing process showed the presence of high levels of calcium sulfate dihydrate (CaSO₄-2H₂O) and low levels of calcium carbonate (CaCO₃) and silicon oxide (SiO₂).



Figure 4.4: XRD of V-B5G

4.4.4.2 X-Ray Fluorescence Analysis of V-B5G

Table 4.9: XRF of V-B5G

Composition (%)	SiO ₂	TiO₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na₂O	K ₂ O	P ₂ O ₅	SO₃	LOI
V-B5G (2017)	0.21	<0.007	<0.022	0.08	0.04	47.85	0.35	0.00	0.00	50.34	1.91
V-B5G (2018)	0.20	0.00	0.06	0.04	0.04	41.00	0.30	0.00	0.00	54.00	-

4.4.5 Chemical Properties of RAF

4.4.5.1 X-Ray Diffraction Analysis of RAF

The mineralogical composition of RAF are plagioclase, calcite, quartz, mica, chlorite and amphibole. The peaks and valleys pattern of the RAF showed the presence of high levels of plagioclase and chlorite followed by moderate levels of calcite and quartz. The amounts of mica and amphibole present in the sample was small. Both chlorite and mica are known as silicate minerals due to their well-defined layers that are formed. The RAF sample exhibits both sedimentary and metamorphic rock properties.

(Coupled TwoTheta/Theta)



Figure 4.5: XRD of RAF

4.4.5.2 X-Ray Fluorescence Analysis of RAF

Table 4.10: XRF of RAF

Composition (%)	SiO ₂	TiO₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P2O5	SO3	MnO
RAF (2017)	39.68	1.44	14.91	10.12	6.60	9.88	3.67	0.90	0.56	2.59	
RAF (2018)	35.00	1.60	12.00	12.00	6.40	12.00	1.90	1.20	0.47	1.30	0.42

4.4.6 Chemical Properties of QWD

4.4.6.1 X-Ray Diffraction Analysis of QWD

The mineralogical composition of QWD are albite, calcite, chlorite and hydrotalcite. The peaks and valleys pattern of QWD showed the presence of moderate levels of albite and calcite. The amounts of chlorite and hydrotalcite present in the sample was small to moderate.



Figure 4.6: XRD of QWD

4.4.6.2 X-Ray Fluorescence Analysis of QWD

Table 4.11: XRF of QWD

Composition (%)	SiO ₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	SO₃	MnO
QWD	34.00	1.80	12.00	12.00	5.30	14.00	2.60	0.87	0.56	0.99	0.41

4.7.7 Chemical Properties of MHT

4.4.7.1 X-Ray Diffraction Analysis of MHT

The mineralogical composition of MHT are portlandite, lime and calcite. The peaks and valleys pattern of the MHT showed the presence of high percentage of portlandite followed by moderate levels of lime and calcite. Both the lime and calcite composed of calcium carbonate (CaCO₃).





Figure 4.7: XRD of MHT

4.4.7.2 X-Ray Fluorescence Analysis of MHT

Table	4.12	XRF	of	MHT

Composition (%)	SiO ₂	TiO₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na₂O	K₂O	P ₂ O ₅	SO₃	LOI
MHT	0.41	0.01	<0.022	0.05	0.70	73.78	<0.013	0.01	0.00	0.17	25.75

4.4.8 Chemical Properties of GGBS

4.4.8.1 X-Ray Fluorescence Analysis of GGBS

Table 4.13: XRF of GGBS obtained from Hanson Heidelberg

Composition (%)	SiO₂	TiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	CaO	Na ₂ O	K₂O	P ₂ O ₅	SO₃	MnO
GGBS (2017)	33.23	1.09	13.14	0.52	8.92	39.76	0.21	0.45	-	1.12	0.33
GGBS (2018)	34.20	0.52	12.62	0.69	6.77	41.12	0.07	0.48	-	1.75	0.31
GGBS (2019)	34.76	0.51	11.44	0.64	7.23	40.80	0.11	0.46	-	1.70	0.30

4.5 Materials Identified to be used in Phase One Mix Design Trial

All the materials sent to Leicester University and the UoW for XRD and XRF analysis had the potential to be used in phase 1 of the experimental design. Based on the chemical analysis and mineralogical properties of all the materials with potential as binders, and to reduce the scope of this research study, LKD and LHT were considered unsuitable for the Phase 1 Mix Design Experiments and were therefore removed from the study due to the lack of alkali contents in their chemical compositions. The lack of alkalis in the mixtures would have therefore resulted to weaker strengths.

4.5.1 Test to Determine the Pozzolanic Properties of Reclaimed Asphalt Filler

In addition to the XRD and XRF analysis carried out on the RAF, further laboratory test was instigated to determine whether the material was pozzolanic. (Neville 2011), formal definition of ASTM 618-08a describes pozzolana as a siliceous or siliceous and aluminous

material which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically react with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties.

The test to determine the pozzolanic activity index of the RAF was carried out in accordance with the requirements set out in (ASTM 2013). In the tests, 20% of RAF and PFA replacement for cement was used. The PFA was used as a reference mix in the experiment.

The experiment revealed that the RAF had over 75% of the strength of the controlled mix at 28 days. Even though it is in line with the requirement test methods as outlined in ASTM C311 which satisfied one of the criteria for determining pozzolanic activity, it was not fully in compliance with the specifications as indicated in ASTM C618 for the second criterium. (Astm 2003) states that to satisfy the pozzolanic requirements, the material should meet the requirements content of pozzolanic oxides (SiO₂+Al₂O₃+Fe₂O₃) of more than 70%. The (SiO₂+Al₂O₃+Fe₂O₃) oxides content of RAF samples tested in 2017 was approximately 65%. The content of the reactive silicon dioxide (SiO₂) shall be not less than 25% by mass stated by (EN, BS 2011). Based on the analysis, the amount of RAF used in this research was considered reactive and acted as pozzolan in the compositions.

4.5.2 Tests to Determine the pH Values of Materials used in Phase One Experiments

pH test was carried out on all the materials used in the Phase 1 experiments. The test was to determine the chemical properties of the samples in regards their acidity and / or alkalinity levels. All the different samples were prepared the same way to achieve consistency in the testing process and for better results comparisons. Each sample weighing twenty grams was placed in a cup and 40 millilitres of water added. The sample and water were mixed and left to saturate for two hours before the test was carried out.

The results of the test revealed that almost all the samples used in the first phase of the experiment had pH values greater than 9. This is equal to the normal pH expected for an alkaline material. Table 4.14 shows the actual values obtained and Figure 4.8 shows photo of the saturated samples and Hanna instrument used.

Material	pH Value				
BPD-C	12.41				
BPD-H	12.76				
SSD (BOS)	12.73				
PG	9.37				
V-B5G	8.63				
RAF	11.68				
QWD	8.27				
MHT	12.12				
GGBS	11.41				

Table 4.14: pH Values of Materials used in the Phase 1 Experiments



Figure 4.8: Saturated Samples ready to be Tested and Hanna Instrument

4.6 Physical Properties of Materials used in Phase One Experiments

4.6.1 Physical Properties of Raw Materials used in the Research

Analysis to determine the physical properties of the raw materials were carried out at Coventry University using sieve sizes ranging from 54 microns to 600 microns and Malvern Mastersize 2000. Figure 4.9 shows the particle size distribution of the raw materials using the sieve sizes. The GGBS, QWD, RAF, V-B5G and BPD-C contain more particles in the range of 54 – 212 microns compared to the other materials. The crushed PG contains more particles ranging between 150 – 300 microns.



Figure 4.9: Particle size distribution of raw materials used in research

4.6.2 Physical Properties of SSD

The physical properties of the SSD were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.10 shows the particle size distribution of the sample. The particles range between 1 to 1000 microns in diameter with an average particle of fine, mid-range and coarse size in the regions of around 0.008, 0.060 and 0.350 mm respectively. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (3.51 g/cm³).



Figure 4.10: Particle size distribution of SSD using Malvern Mastersize 2000

4.6.3 Physical Properties of PG

The physical properties of the PG were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.11 shows the particle size distribution of the sample. The particles range between 1.5 to 1500 microns in diameter with an average particle size in the region of around 0.300 mm. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (2.31 g/cm³).



Figure 4.11: Particle size distribution of PG using Malvern Mastersize 2000

4.6.4 Physical Properties of V-B5G

The physical properties of the V-B5G were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.12 shows the particle size distribution of the sample. The particles range between 0.5 to 1300 microns in diameter with an average particle size in the region of around 0.100 mm. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (2.35 g/cm³).



Figure 4.12: Particle size distribution of V-B5G using Malvern Mastersize 2000

4.6.5 Physical Properties of RAF

The physical properties of the RAF were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.13 shows the particle size distribution of the sample. The particles range between 1.3 to 350 microns in diameter with an average particle size in the region of around 0.028 mm. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (2.71 g/cm³).



Figure 4.13: Particle size distribution of RAF using Malvern Mastersize 2000

4.6.6 Physical Properties of QWD

The physical properties of the QWD were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.14 shows the particle size distribution of the sample. The particles range between 1.3 to 400 microns in diameter with an average particle size in the region of around 0.037 mm. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (2.66 g/cm³).



Figure 4.14: Particle size distribution of QWD using Malvern Mastersize 2000

4.6.7 Physical Properties of MHT

The physical properties of the MHT were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.15 shows the particle size distribution of the sample. The particles range between 1.5 to 2000 microns in diameter with an average particle of fine and coarse size in the region of around 0.060 and 0.500 mm respectively.



Figure 4.15: Particle size distribution of MHT using Malvern Mastersize 2000

4.6.8 Physical Properties of GGBS

The physical properties of the GGBS were determined using a Malvern Mastersize 2000 and the outputs created by the Malvern Laser Granulometer with an accuracy of +/-1%. Figure 4.16 shows the particle size distribution of the sample. The particles range between 0.3 to 130 microns in diameter with an average particle size in the region of around 0.045 mm. AccuPyc 1330 Gas Helium Pycnometer was used to determine the specific gravity of the material (2.87 g/cm³).



Figure 4.16: Particle size distribution of GGBS using Malvern Mastersize 2000 – *Awaiting Results*

CHAPTER 5 – PHASE ONE EXPERIMENTAL PROGRAMME

5.1 Introduction

This chapter explains how the experiment was designed and the samples prepared. The mixing, compacting, casting and curing procedures used to undertake this experiment are explained in detailed.

5.2 Experiment Strategy of Research Study

5.2.1 Design and Optimisation of the Cementitious Binders

The Design of Experiment (DoE) was carried out using a statistical mixture design programme – Minitab 18. Eleven combinations of ternary binders referred to as in this study 'Group' were designed, analysed and the preferred groups that would be used for the phase two experiment. The Extreme Vertices Design (EVD) method was used to set the boundaries of the components in each Group for the designs.

These materials used in this research study were analysed in terms of their mechanical stability and their performance determined in the strength development. The best composition of these materials was determined by carrying out compressive strength tests at 3, 7, 14, 28, 90 and 180 days of the materials in different proportions using 50mm cubes.

5.2.2 Selecting the Correct Mixture Design – Minitab

There are four types of mixture designs in Minitab: i) Simplex Centroid; ii) Simplex Lattice – 1st Degree; iii) Simplex Lattice – 2nd Degree; and iv) Extreme Vertices – 2nd Degree. All four can be designed either augmented or unaugmented. Augmented design is whereby design points are included in the interior of the design space, unaugmented on the other hand do not with the exception of Simplex Centroid design. The 1st Degree only allows the design to look for linear effect. The 2nd Degree on the other hand allows the design to find quadratics.

Even though Simplex Lattice – 2^{nd} Degree and Extreme Vertices – 2^{nd} Degree permit fitting up to a quadratic model, Extreme Vertices design is appropriate for the relationships the author was looking for. The Extreme Vertices design was chosen for this experimental

study because of the provisions of the upper and lower bounds used in the ternary combinations, it allows precision in the design, provides better estimates, maximises variance and generates plots for all three components in the model. This allows for good coverage and the design points adequately covers the design space. The unique design space on Extreme Vertices designs allow points to be placed not only at the centroid, midpoints and axial points, but also at the extreme vertices of the region.

The following questions are answered when using Extreme Vertices designs:

- it satisfies the formulation in having constraints that ensure correct specifications.
 For example, which of the ternary sample(s) that satisfy the pozzolanic effect in the mixture and at what limit (upper – lower);
- it satisfies the formulation that optimizes the response for certain reaction. For instance, which of the sample(s) that maximizes alkalinity in the mixture; and
- it satisfies the formulation on how the different samples used in the ternary groups affect a response. For example, which of the sample that maximizes compressive strength in the mixture.

Guidance on the lower / upper boundaries used for some of the ternary combinations used in this research study were taken from literatures carried out by other researchers using similar materials.

5.3 Preparation of Samples used in Phase One Experiments

5.3.1 By-pass Dust (BPD-C) Preparation

Preparation of the BPD-C received from Cemex was not required. The sample was delivered dry and particle size less than 600 microns therefore, grinding of the sample was not required.

5.3.2 By-pass Dust (BPD-H) Preparation

The BPD-H received from Hanson Cement required preparation before use. The sample was delivered semi-dry and some of the materials were in a form of pellets. The sample was put in the oven at 50°C and left to dry for 48 to 72 hours. The particle size of the sample was less than 600 microns and therefore grinding of the sample was not required.

5.3.3 Steel Slag Dust (SSD) Preparation

The SSD was delivered moist and had to be put in an oven at 50°C and left to dry for 48 to 72 hours. Sieving of the sample passing 600 microns was carried out. Two-third of the cylindrical barrels containing steel balls of 12 mm, 20 mm and 25 mm in size were filled with the sample of particle size greater than 600 microns. The ball milling machine was switched on and left to grind for 72 to 96 hours. The reduction in sample size was the combination of the grinding process within the cylindrical barrels caused by the rotation of the barrels around a horizontal axis at a speed of 0.8 cycles per seconds.

Further sieving, passing 600 microns of the sample was carried out and the ball milling process repeated until all the sample had passed through a 600 microns sieve. Figure 5.1 shows the ball milling machine used to grind the sample.



Figure 5.1: Ball Milling Machine

To maintain consistency with the grading of the sample, all the sieved sample was placed in a concrete mixer (capacity 100 litres) and left to rotate for three minutes.
5.3.4 Plasterboard Gypsum (PG) Preparation

The plasterboards were broken into sizable pieces of approximately 600mm by 500mm. The plasterboards were further broken down into smaller pieces and were grounded to remove the paper backing. This grinding exercise was done by using different sieve sizes to fully de-contaminate the plasterboard gypsum of paper. Figure 5.2 shows the PG being broken down into sizable pieces and Figure 5.3 shows a photo of the plasterboard gypsum when sieved to remove the paper backing.



Figure 5.2: Plasterboard broken down into sizable pieces



Figure 5.3: Sieving of plasterboard to remove paper

After all the paper backing has been completely removed, it was further grind to a fine powder passing through a 600 microns sieve.

5.3.5 Vitamin B5 Gypsum (V-B5G) Preparation

The raw sample was delivered wet and had to be treated before use. The wet sample was placed in a tray and put in an oven at 30°C and left to dry for 72 to 96 hours. Sieving of the sample was not required as the largest particle size was approximately 140 microns.

After the sample was left to dry and cool down, it was tested for its moisture contents and the correct moisture contents were factored into the mix designs. Both the quantity of the wet sample and that of the actual water required were taken into consideration during the mixing process.

5.3.6 Reclaimed Asphalt Filler (RAF) Preparation

Preparation of the reclaimed asphalt filler received from Tarmac was not required. The sample was delivered dry and particle size between 0 to 63 microns.

5.3.7 Quarry Waste Dust (QWD) Preparation

Preparation of the quarry waste dust received from Tarmac was not required. The sample was delivered dry and particle size less than 600 microns.

5.3.8 Medium Hydrated Tailing (MHT) Preparation

The sample received from Tarmac was dry, in a powder / granular format and therefore required sieving as 90% of the particle size passed through a 600 microns sieve.

5.3.9 Ground Granulated Blast Furnace Slag (GGBS) Preparation

Preparation of the GGBS was not required. The sample was delivered dry and the particle size less than 600 microns. This material was part of Coventry University material inventory stock. It was sourced from Hanson Heidelberg Cement Group and delivered from their Teesside Plant.

5.4 Mixing of Binder Samples

The mixing of the binder samples was carried out in a KENWOOD mechanical mixer of 2-litres capacity. The following procedures were used to carry out the mixing exercise:

- the samples were mixed dry for 1 minute with the rotating blade set at level 1 speed;
- ii. whilst the blade was still rotating at level 1 speed, half of the water was added and the mixing continued for another 30 seconds;
- iii. the speed of the rotating blade was increased to level 2 and the mixing continued for further 30 seconds;

- iv. the mixer was stopped at 2 minutes of mixing, the blade and side / bottom of the mixing bowl was scrapped off to ensure all the samples have been mixed properly for 1 minute;
- v. the mixer was switched-on and mixing of the sample continued for 2 minutes at level 3 speed whilst the remaining water was slowly added;
- vi. after the mixer was switched-off, mixing of the sample continued by hand for 1 minute using spatula.

The total time taken for the whole mixing exercise was approximately 6 minutes. The KENWOOD mixer used for this exercise is shown in Figure 5.4.



Figure 5.4: KENWOOD machine with sample in the bowl ready to be mixed

5.5 Weighing and Compaction of Samples

An alternative method of compaction to that specified in (BS EN 12390 - 2 2000) was used in this laboratory study to achieve a better density as it was a semi-dry paste. The sample was pre-compacted with a square tamping rod (25mm x 25mm x 300mm) in 3 layers of 90g (270g total cube mass) per cube at 25 strokes per layer. Figures 5.5 and

5.6 show the plastic containers used to weigh the 90g samples and the 50mm cube moulds (gang of three) being prepared for use respectively.



Figure 5.5: Weighing scale and plastic containers used to place 90g sample



Figure 5.6: Gang of three 50mm cube moulds being prepared for use

The Avery Denison 2000 machine was used to hydraulically press the samples. The pressure was not manually controlled, the machine was linked to a desk top computer and the hydraulic pressure was set at 90KN for a gang of three 50mm cube moulds prepared.



Figure 5.7: Gang of three 50mm mould compacted at 90KN



Figure 5.8: Avery Denison 2000 machine linked to a computer

The 90KN pressure was set to simulate real life application in construction site environment. A constant rate of loading at 5KN/s was applied to the samples. The hydraulic compaction of 30KN per cube pressure used in this research study is in line with the requirements set out in SHW (2020) MCHW 800 Series Clause 813. This is whereby either a vibrating or pneumatic-tyred roller with a wheel loading of not less than 30KN followed by at least eight passes is used to compact hydraulically bound mixtures. The pressure of 90KN load was applied to the sample for 2 minutes before it was released. Figures 5.7 and 5.8 show a gang of three 50mm mould compacted at 90KN and the Avery Denison machine linked to a desk top computer.

5.6 Casting and Curing of Test Specimens

The 50mm semi-dry specimens were easy to handle and therefore casting was carried out immediately after the specimens had been hydraulically pressed without a shock or vibration. The specimens were cured in transparent propagators at 20° C +/- 2° C with a relative humidity of between 95 – 98%. Figures 5.9 and 5.10 show the specimens in the propagators being cured.



Figure 5.9: Specimens being cured in propagator



Figure 5.10: Label showing production dates mixes were prepared

5.7 Testing of Specimens

Two different testing machines were used to determine the compressive strengths of the 50 mm cube paste specimens. The JJ Lloyd – 50 computerised testing equipment was used for specimens expected to achieve a maximum failure load of up to 50,000N. The loading rate was set constant at 5 mm/min and the results reported in N/mm2 or MPa. Compressive strength test of the hardened paste was based on (BS EN 12390 - 3 2002). Figure 5.11 shows the JJ Lloyd – 50 equipment in use.



Figure 5.11: JJ Lloyd – 50 Equipment linked to a computer

For paste specimens expected to achieve load on failure greater than 50,000N, the Avery Denison 2000 cube crusher was used. The cube crusher has a maximum load of 2000KN and the loading rate was set at 5KN/sec. The machine was linked to a desk top computer where all the data was collected and saved. Figure 5.12 shows the Avery Denison 2000 machine in use.



Figure 5.12: Avery Denison 2000 machine linked to a computer

CHAPTER 6 – PHASE ONE AND PHASE TWO EXPERIMENTAL TESTS AND RESULTS

6.1 Introduction

This chapter explains the different groups that were used in this research study and their design constraints. It expands on the Phase One mix design / results and details the Phase Two experimental work in identifying the chosen groups with potentials. It provides the simplex design plot and results obtained from Minitab for the 7, 28, 90 and 180 days compressive strength in producing the 50mm hydraulic binder cubes.

6.2 Mix Designs of Cement Binders

6.2.1 Group 1 – PG/MHT/GGBS

The ternary combination analysed for Group 1 was PG, MHT and GGBS. The upper limit boundaries used for PG, MHT and GGBS were 80%, 15% and 60% by weight respectively. The lower limit boundaries were 25% for PG, 0% for MHT and 5% for GGBS. Detailed in Figure 6.1 is the simplex design plot produced by Minitab. Table 6.1 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components							Mix De	esign					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
PG	80	40	68.13	32.5	80	25	80	48.13	56.25	60	68.13	52.5	40.63
МНТ	0	0	3.75	7.5	7.5	15	15	3.75	7.5	0	11.25	15	11.25
GGBS	20	60	28.13	60	12.5	60	5	48.13	36.25	40	20.63	32.5	48.13

Table 6.1: Group 1 – Mix Design (PG/MHT/GGBS)



Figure 6.1: Group 1 – Simplex Design Plot (PG/MHT/GGBS)

6.2.2 Group 2 – PG/RAF/GGBS

The ternary combination analysed for Group 2 was PG, RAF and GGBS. The upper limit boundaries used for PG, RAF and GGBS were 20%, 80% and 60% by weight respectively. The lower limit boundaries were 0% for PG, 20% for RAF and 0% for GGBS. Detailed in Figure 6.2 is the simplex design plot produced by Minitab. Table 6.2 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						N	lix Desig	gn					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
PG	20	10	10	5	20	15	20	0	15	0	0	5	10
RAF	20	80	55	47.5	50	37.5	80	80	67.5	40	60	67.5	30
GGBS	60	10	35	47.5	30	47.5	0	20	17.5	60	40	27.5	60

Table 6.2: Group 2 – Mix Design (PG/RAF/GGBS)



Figure 6.2: Group 2 – Simplex Design Plot (PG/RAF/GGBS)

6.2.3 Group 2A - V-B5G/RAF/GGBS

The ternary combination analysed for Group 2A was V-B5G, RAF and GGBS. The upper limit boundaries used for V-B5G, RAF and GGBS were 20%, 80% and 60% by weight respectively. The lower limit boundaries were 0% for V-B5G, 20% for RAF and 0% for GGBS. Detailed in Figure 6.3 is the simplex design plot produced by Minitab. Table 6.3 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						Μ	lix Desig	gn					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	20	10	10	5	20	15	20	0	15	0	0	5	10
RAF	20	80	55	47.5	50	37.5	80	80	67.5	40	60	67.5	30
GGBS	60	10	35	47.5	30	47.5	0	20	17.5	60	40	27.5	60

Table 6.3: Group 2A – Mix Design (V-B5G/RAF/GGBS)



Figure 6.3: Group 2A – Simplex Design Plot (V-B5G/RAF/GGBS)

6.2.4 Group 3 – PG/GGBS/BPD-C

The ternary combination analysed for Group 3 was PG, GGBS and BPD-C. The upper limit boundaries used for PG, GGBS and BPD-C were 60%, 80% and 10% by weight respectively. The lower limit boundaries were 10% for PG, 30% for GGBS and 0% for BPD-C. Detailed in Figure 6.4 is the simplex design plot produced by Minitab. Table 6.4 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						I	Mix Desig	gn					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
PG	48.75	40	60	15	37.5	35	28.75	60	48.75	23.75	10	60	20
GGBS	43.75	60	40	80	57.5	55	68.75	35	48.75	68.75	80	30	80
BPD-C	7.5	0	0	5	5	10	2.5	5	2.5	7.5	10	10	0

Table 6.4: Group 3 – Mix Design (PG/GGBS/BPD-C)



Figure 6.4: Group 3 – Simplex Design Plot (PG/GGBS/BPD-C)

6.2.5 Group 4 – PG/GGBS/BPD-H

The ternary combination analysed for Group 4 was PG, GGBS and BPD-H. The upper limit boundaries used for PG, GGBS and BPD-H were 60%, 80% and 15% by weight respectively. The lower limit boundaries were 5% for PG, 25% for GGBS and 0% for BPD-H. Detailed in Figure 6.5 is the simplex design plot produced by Minitab. Table 6.5 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

13

32.5

52.5

15

Components						N	lix Desig	gn				
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12
PG	5	40	60	48.13	60	36.25	48.13	20.63	60	12.5	28.13	20
GGBS	80	60	40	40.63	25	56.25	48.13	68.13	32.5	80	68.13	80
BPD-H	15	0	0	11.25	15	7.5	3.75	11.25	7.5	7.5	3.75	0

Table 6.5: Group 4 – Mix Design (PG/GGBS/BPD-H)



Figure 6.5: Group 4 – Simplex Design Plot (PG/GGBS/BPD-H)

6.2.6 Group 5 – V-B5G/MHT/SSD

The ternary combination analysed for Group 5 was V-B5G, MHT and SSD. The upper limit boundaries used for V-B5G, MHT and SSD were 80%, 15% and 60% by weight respectively. The lower limit boundaries were 25% for V-B5G, 0% for MHT and 5% for SSD. Detailed in Figure 6.6 is the simplex design plot produced by Minitab. Table 6.6 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components							Mix I	Design					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	40	68.125	25	80	60	32.5	80	48.125	68.125	40.625	52.5	56.25	80
MHT	0	3.75	15	0	0	7.5	15	3.75	11.25	11.25	15	7.5	7.5
SSD	60	28.125	60	20	40	60	5	48.125	20.625	48.125	32.5	36.25	12.5

Table 6.6: Group 5 – Mix Design (V-B5G/MHT/SSD)



Figure 6.6: Group 5 – Simplex Design Plot (V-B5G/MHT/SSD)

6.2.7 Group 6 - V-B5G/RAF/SSD

The ternary combination analysed for Group 6 was V-B5G, RAF and SSD. The upper limit boundaries used for V-B5G, RAF and SSD were 20%, 80% and 60% by weight respectively. The lower limit boundaries were 0% for V-B5G, 20% for RAF and 0% for SSD. Detailed in Figure 6.7 is the simplex design plot produced by Minitab. Table 6.7 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Table 6.7: Group 6 – Mix Design (V-B5G/RAF/SSD)

Components						N	lix Desi	gn					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	20	0	20	0	15	0	5	5	10	20	10	10	15
RAF	20	40	80	60	37.5	80	47.5	67.5	55	50	80	30	67.5
SSD	60	60	0	40	47.5	20	47.5	27.5	35	30	10	60	17.5



Figure 6.7: Group 6 – Simplex Design Plot (V-B5G/RAF/SSD)

6.2.8 Group 7 - V-B5G/SSD/BPD-C

The ternary combination analysed for Group 7 was V-B5G, SSD and BPD-C. The upper limit boundaries used for V-B5G, SSD and BPD-C were 60%, 80% and 15% by weight respectively. The lower limit boundaries were 5% for V-B5G, 25% for SSD and 0% for BPD-C. Detailed in Figure 6.8 is the simplex design plot produced by Minitab. Table 6.8 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						Mix	Design						
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	36.25	20.63	32.5	28.13	5	48.13	48.13	60	60	60	40	20	12.5
SSD	56.25	68.13	52.5	68.13	80	48.13	40.63	25	32.5	40	60	80	80
BPD-C	7.5	11.25	15	3.75	15	3.75	11.25	15	7.5	0	0	0	7.5

Table 6.8: Group 7 – Mix Design (V-B5G/SSD/BPD-C)



Figure 6.8: Group 7 – Simplex Design Plot (V-B5G/SSD/BPD-C)

6.2.9 Group 8 – V-B5G/SSD/BPD-H

The ternary combination analysed for Group 8 was V-B5G, SSD and BPD-H. The upper limit boundaries used for V-B5G, SSD and BPD-H were 60%, 80% and 15% by weight respectively. The lower limit boundaries were 5% for V-B5G, 25% for SSD and 0% for BPD-H. Detailed in Figure 6.9 is the simplex design plot produced by Minitab. Table 6.9 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						Mix C	Design						
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	36.25	20.625	32.5	28.125	5	48.125	48.125	60	60	60	40	20	12.5
SSD	56.25	68.125	52.5	68.125	80	48.125	40.625	25	32.5	40	60	80	80
BPD-H	7.5	11.25	15	3.75	15	3.75	11.25	15	7.5	0	0	0	7.5

Table 6.9: Group 8 – Mix Design (V-B5G/SSD/BPD-H)



Figure 6.9: Group 8 – Simplex Design Plot (V-B5G/SSD/BPD-H)

6.2.10 Group 9 - PG/QWD/GGBS

The ternary combination analysed for Group 9 was PG, QWD and GGBS. The upper limit boundaries used for PG, QWD and GGBS were 20%, 80% and 60% by weight respectively. The lower limit boundaries were 0% for PG, 20% for QWD and 0% for GGBS. Detailed in Figure 6.10 is the simplex design plot produced by Minitab. Table 6.10 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Components						Mi	x Desi	gn					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
PG	20	10	10	5	20	15	20	0	15	0	0	5	10
QWD	20	80	55	47.5	50	37.5	80	80	67.5	40	60	67.5	30
GGBS	60	10	35	47.5	30	47.5	0	20	17.5	60	40	27.5	60

Table 6.10: Group 9 – Mix Design (PG/QWD/GGBS)



Figure 6.10: Group 9 – Simplex Design Plot (PG/QWD/GGBS)

6.2.11 Group 9A – V-B5G/QWD/GGBS

The ternary combination analysed for Group 9A was V-B5G, QWD and GGBS. The upper limit boundaries used for V-B5G, QWD and GGBS were 20%, 80% and 60% by weight respectively. The lower limit boundaries were 0% for V-B5G, 20% for QWD and 0% for GGBS. Detailed in Figure 6.11 is the simplex design plot produced by Minitab. Table 6.11 shows the proportion of the materials required in each mix. It was assumed that due to the high levels of materials used in some of the components, the combination of binary and ternary mixes was produced in the mix design.

Table 6.11:	Group 9A -	Mix Design	(V-B5G/QWD/GGBS)

Components						М	ix Des	ign					
(% wt)	1	2	3	4	5	6	7	8	9	10	11	12	13
V-B5G	20	10	10	5	20	15	20	0	15	0	0	5	10
QWD	20	80	55	47.5	50	37.5	80	80	67.5	40	60	67.5	30
GGBS	60	10	35	47.5	30	47.5	0	20	17.5	60	40	27.5	60



Figure 6.11: Group 9A – Simplex Design Plot (V-B5G/QWD/GGBS)

6.3 Compressive Strength Results

- 6.3.1 Group 1 Mixture Design Results
- 6.3.1.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the PG/MHT/GGBS binder mixes are shown in Table 6.12. Mix 2, with the proportions of 40% PG and 60% GGBS attained the highest compressive strength in Group 1. The maximum value achieved was 16MPa at 28 days.

Table 6.12: Group 1 – Compressive Strength (PG/MHT/GGBS)

Mix Design Deference	Strength at	Days (MPa)		
wix Design Reference	7	28	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - PG80/GGBS20	3	3	0.15	1865
Mix 2 - PG40/GGBS60	3	16*	0.15	1943
Mix 3 - PG68.125/MHT3.75/GGBS28.125	5	6	0.15	1712
Mix 4 - PG32.50/MHT7.50/GGBS60	7	13	0.15	1858
Mix 5 - PG80/MHT7.50/GGBS12.50	4	5	0.15	1692
Mix 6 - PG25/MHT15/GGBS60	8	10	0.15	1795
Mix 7 - PG80/MHT15/GGBS5	2	3	0.15	1666
Mix 8 - PG48.125/MHT3.75/GGBS48.125	6	8	0.15	1778
Mix 9 - PG56.25/MHT7.50/GGBS36.25	5	6	0.15	1742
Mix 10 - PG60/GGBS40	2	8	0.15	1842
Mix 11 - PG68.125/MHT11.25/GGBS20.625	4	4	0.15	1658
Mix 12 - PG52.50/MHT15/GGBS32.50	5	6	0.15	1662
Mix 13 - PG40.625/MHT11.25/GGBS48.125	6	8	0.15	1722
* Highlights highest compressive strength in	n the group			

6.3.1.2 Contour Plots for 7 and 28 Days Compressive Strength

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.12 and 6.13 respectively.



Figure 6.12: Group 1 – 7 Days Compressive Strength Contour Plot

Group 1 - Contour Plot



Figure 6.13: Group 1 – 28 Days Compressive Strength Contour Plot

6.3.1.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength are shown in Figure 6.14. Mixes 1 (PG80/GGBS20) and 11 (PG68.125/MHT11.25/GGBS20.625) of this Group showed a decrease in strength as the specimen ages. There was a significant increase in strength for Mix 2 (PG40/GBF60) of approximately 400% from the 7 days strength.



Figure 6.14: Comparisons of 7 and 28 Days Compressive

6.3.2 Group 2 Mixture Design Results

6.3.2.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the PG/RAF/GGBS binder mixes are shown in Table 6.13. Mix 4, with the proportions of 5% PG and 47.5% RAF and 47.5% GGBS attained the highest compressive strength in Group 2. The maximum value achieved was 27MPa at 28 days.

Mix Design Deference	Strength at	Days (MPa)	L/C Datia	Dry Density (Kg/m3)
Wix Design Reference	7	28	L/S Ratio	
Mix 1 - PG20/RAF20/GGBS60	2	24	0.15	1821
Mix 2 - PG10/RAF80/GGBS10	1	2	0.15	2370
Mix 3 - PG10/RAF55/GGBS35	1	24	0.15	2034
Mix 4 - PG5/RAF47.50/GGBS47.50	1	27*	0.15	2002
Mix 5 - PG20/RAF50/GGBS30	1	14	0.15	1984
Mix 6 - PG15/RAF37.50/GGBS47.50	1	25	0.15	1965
Mix 7 - PG20/RAF80	1	1	0.15	2293
Mix 8 - RAF80/GGBS20	1	3	0.15	2198
Mix 9 - PG15/RAF67.50/GGBS17.50	1	11	0.15	2052
Mix 10 - RAF40/GGBS60	1	4	0.15	1980
Mix 11 - RAF60/GGBS40	1	3	0.15	2072
Mix 12 - PG5/RAF67.50/GGBS27.50	2	23	0.15	2110
Mix 13 - PG10/RAF30/GGBS60	1	26	0.15	1951
* Highlights highest compressive strength i	n the group			

Table 6.13: Group 2 – Compressive Strength (PG/RAF/GGBS)

6.3.2.2 Contour Plots for 7 and 28 Days Compressive Strength

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.15 and 6.16 respectively.



Figure 6.15: Group 2 – 7 Days Compressive Strength Contour Plot



Figure 6.16: Group 2 – 28 Days Compressive Strength Contour Plot

6.3.2.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength are shown in Figure 6.17. All the mixes in this composition showed a significant increase in strength development as the specimen ages except for Mix 7 (PG20/RAF80). The increase in the average compressive strength from 7 to 28 days for the top six mixes exceeding 20MPa is approximately 1,500%.



Figure 6.17: Comparisons of 7 and 28 Days Compressive Strength

6.3.3 Group 2A Mixture Design Results

6.3.3.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the V-B5G/RAF/GGBS binder mixes are shown in Table 6.14. Mix 6, with the proportions of 15% V-B5G and 37.5% RAF and 47.5% GGBS attained the highest compressive strength in Group 2A. The maximum value achieved was 29MPa. The results of the liquid / solid ratios of the mixes in Group 2A were affected because the moisture content of the V-B5G material was factored in the mix design calculations retrospectively of the laboratory experiments. The moisture content present in the V-B5G has mainly two effects in the mix design: i) it reduces the water needed in the original mix design; and ii) varies the proportion by increasing the dry raw material needed for the experiment. In other words, a higher percentage of moisture content in the raw material leads to smaller amount of water and an increase in the wet sample in the mix design.

	Strength at	t Days (MPa)		
IVIIX Design Reference	7	28	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - V-B5G20/RAF20/GGBS60	3	25	21.0	1930
Mix 2 - V-B5G10/RAF80/GGBS10	1	4	18.0	2400
Mix 3 - V-B5G10/RAF55/GGBS35	1	26	18.0	2074
Mix 4 - V-B5G5/RAF47.50/GGBS47.50	1	24	16.5	2020
Mix 5 - V-B5G20/RAF50/GGBS30	2	22	21.0	2025
Mix 6 - V-B5G15/RAF37.50/GGBS47.50	1	29*	19.5	1982
Mix 7 - V-B5G20/RAF80	1	0	21.0	2408
Mix 8 - RAF80/GGBS20	1	3	15.0	2198
Mix 9 - V-B5G15/RAF67.50/GGBS17.50	1	13	19.5	2091
Mix 10 - RAF40/GGBS60	1	4	15.0	1973
Mix 11 - RAF60/GGBS40	1	3	15.0	2071
Mix 12 - V-B5G5/RAF67.50/GGBS27.50	1	22	16.5	2169
Mix 13 - V-B5G10/RAF30/GGBS60	2	28	18.0	2007
* Highlights highest compressive strength in	n the group			

Table 6.14: Group 2A – Compressive Strength (V-B5G/RAF/GGBS)

6.3.3.2 Contour Plots for 7 and 28 Days Compressive Strength

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.18 and 6.19 respectively.



Figure 6.18: Group 2A – 7 Days Compressive Strength Contour Plot



Group 2A - Contour Plot (28 Day Strength - V-B5G/RAF/GGBS)

Figure 6.19: Group 2A – 28 Days Compressive Strength Contour Plot

6.3.3.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength

are shown in Figure 6.20. All the mixes in this composition showed a significant increase in strength development as the specimen ages except for Mix 7 (V-B5G20/RAF80). The increase in the average compressive strength from 7 to 28 days for the top seven mixes exceeding 20MPa is approximately 1,500%.



Figure 6.20: Comparisons of 7 and 28 Days Compressive Strength

6.3.4 Group 3 Mixture Design Results

6.3.4.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the PG/GGBS/BPD-C binder mixes are shown in Table 6.15. Mix 11, with the proportions of 10% PG, 80% GGBS and 10% of BPD-C attained the highest compressive strength in Group 3. The maximum value achieved was 22MPa.

Mix Design Reference	Strength at	Days (MPa)	L/C Datia		
wix Design Reference	7	28	L/S Ratio	Dry Density (Kg/113)	
Mix 1 - PG48.75/GGBS43.75/BPD-C7.50	8	8	0.15	1770	
Mix 2 - PG40/GGBS60	3	16	0.15	1943	
Mix 3 - PG60/GGBS40	2	8	0.15	1787	
Mix 4 - PG15/GGBS80/BPD-C5	15	19	0.15	1710	
Mix 5 - PG37.50/GGBS57.50/BPD-C5	13	15	0.15	1694	
Mix 6 - PG35/GGBS55/BPD-C10	8	10	0.15	1475	
Mix 7 - PG28.75/GGBS68.75/BPD-C2.50	15	19	0.15	1660	
Mix 8 - PG60/GGBS35/BPD-C5	5	8	0.15	1534	
Mix 9 - PG48.75/GGBS48.75/BPD-C2.50	14	18	0.15	1700	
Mix 10 - PG23.75/GGBS68.75/BPD-C7.50	13	17	0.15	1738	
Mix 11 - PG10/GGBS80/BPD-C10	15	22*	0.15	1778	
Mix 12 - PG60/GGBS30/BPD-C10	7	8	0.15	1611	
Mix 13 - PG20/GGBS80	3	19	0.15	1755	
* Highlights highest compressive strength ir	n the group				

Table 6.15: Group 3 – Compressive Strength (PG/GGBS/BPD-C)

6.3.4.2 Contour Plots for 7 and 28 Days Compressive Strength

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.21 and 6.22 respectively.



Figure 6.21: Group 3 – 7 Days Compressive Strength Contour Plot



Figure 6.22: Group 3 – 28 Days Compressive Strength Contour Plot

6.3.4.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength are shown in Figure 6.23. The mixes in this composition showed an increase in strength development as the specimen ages. All the mixes except for Mix 1 (PG48.75/GGBS43.75/BPD-C7.5) showed an increase in strength development. Mixes 2 and 13 however, showed an increase of approximately 400% from the 7 days strength.



Figure 6.23: Comparisons of 7 and 28 Days Compressive Strength

6.3.5 Group 4 Mixture Design Results

6.3.5.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the PG/GGBS/BPD-H binder mixes are shown in Table 6.16. Mixes 1 and 8 with the proportions of 5%PG, 80%GGBS, 15%BPD-H and 20.625%PG, 68.125%GGBS, 11.25%BPD-H respectively attained the highest compressive strength in Group 4. The maximum value achieved was 25MPa.

Min Design Defenses	Strength at	Days (MPa)		Drus Danaitus (Kalma)
Mix Design Reference	7	28	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - PG5/GGBS80/BPD-H15	17	25*	0.15	1883
Mix 2 - PG40/GGBS60	3	16	0.15	1943
Mix 3 - PG60/GGBS40	2	8	0.15	1842
Mix 4 - PG48.125/GGBS40/BPD-H11.25	9	16	0.15	1835
Mix 5 - PG60/GGBS25/BPD-H15	7	9	0.15	1801
Mix 6 - PG36.25/GGBS56.25/BPD-H7.50	15	21	0.15	1813
Mix 7 - PG48.125/GGBS48.125/BPD-H3.75	13	21	0.15	1754
Mix 8 - PG20.625/GGBS68.125/BPD-H11.25	17	25*	0.15	1841
Mix 9 - PG60/GGBS32.50/BPD-H7.50	10	13	0.15	1722
Mix 10 - PG12.50/GGBS80/BPD-H7.50	15	22	0.15	1805
Mix 11 - PG28.125/GGBS68.125/BPD-H3.75	17	23	0.15	1780
Mix 12 - PG20/GGBS80	3	19	0.15	1755
Mix 13 - PG32.50/GGBS52.50/BPD-H15	10	16	0.15	1842
* Highlights highest compressive strength in	the group			

Table 6.16: Group 4 – Compressive Strength (PG/GGBS/BPD-H)

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6.3.5.2 Contour Plots for 7 and 28 Days Compressive Strength

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.24 and 6.25 respectively.



Figure 6.24: Group 4 – 7 Days Compressive Strength Contour Plot



Group 4 - Contour Plot 28 Day Strength - (PG/GGBS/BPD-H)

Figure 6.25: Group 4 – 28 Days Compressive Strength Contour Plot

6.3.5.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength are shown in Figure 6.26. All the mixes in this composition showed an increase in strength development as the specimen ages.



Figure 6.26: Comparisons of 7 and 28 Days Compressive Strength

- 6.3.6 Group 5 Mixture Design Results
- 6.3.6.1 Mix Proportions and Compressive Strengths

The results for the 7 and 28 days compressive strength tests for the V-B5G/MHT/SSD binder mixes are shown in Table 6.17. Mix 1, with the proportions of 40% V-B5G, 60% SSD attained the highest compressive strength in Group 5. The maximum value achieved was 10MPa.

Table 6.17: Group	5 – Compressive	Strength	(V-B5G/MHT/SSD)
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	Strength at	Days (MPa)						
Mix Design Reference	7	28	L/S Ratio	Dry Density (Kg/m3)				
Mix 1 - V-B5G40/SSD60	4	10*	0.15	2188				
Mix 2 - V-B5G68.125/MHT3.75/SSD28.125	1	2	0.15	1838				
Mix 3 - V-B5G25/MHT15/SSD60	1	2	0.15	2032				
Mix 4 - V-B5G80/SSD20	1	1	0.15	1806				
Mix 5 - V-B5G60/SSD40	2	3	0.15	1948				
Mix 6 - V-B5G32.50/MHT7.50/SSD60	2	4	0.15	2078				
Mix 7 - V-B5G80/MHT15/SSD5	0	1	0.15	1673				
Mix 8 - V-B5G48.125/MHT3.75/SSD48.125	3	5	0.15	1977				
Mix 9 - V-B5G68.125/MHT1.25/SSD20.625	1	1	0.15	1711				
Mix 10 - V-B5G40.625/MHT11.25/SSD48.125	1	2	0.15	1912				
Mix 11 - V-B5G52.50/MHT15/SSD32.50	0	1	0.15	1799				
Mix 12 - V-B5G56.25/MHT7.50/SSD36.25	1	2	0.15	1862				
Mix 13 - V-B5G80/MHT7.50/SSD12.50	1	1	0.15	1751				
* Highlights highest compressive strength in the group								

6.3.6.2 Contour Plots for 7 and 28 Days Compressive Strength

Group 5 - Contour Plot

The response data for the 7 and 28 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.27 and 6.28 respectively.

(7 Day Strength - V-B5G/MHT/SSD) V-B5G Strength (MPa) < 0 0 -2 2 _ 4 4 6 6 _ 8 > 8 0 0 MHT SSD

Figure 6.27: Group 5 – 7 Days Compressive Strength Contour Plot



Figure 6.28: Group 5 – 28 Days Compressive Strength Contour Plot

6.3.6.3 Comparisons of 7 and 28 Days Compressive Strength

Comparisons of the test results obtained from the 7 and 28 days compressive strength are shown in Figure 6.29. All the mixes in this composition showed an increase in strength development as the specimen ages except mixes 4 and 9.



Figure 6.29: Comparisons of 7 and 28 Days Compressive Strength

- 6.3.7 Group 6 Mixture Design Results
- 6.3.7.1 Mix Proportions and Compressive Strengths

The results for the 7, 28 and 90 days compressive strength tests for the V-B5G/RAF/SSD binder mixes are shown in Table 6.18. Mixes 2 and 7 with the proportions of 40%RAF, 60%SSD and 5%V-B5G, 47.5%RAF, 47.5%SSD respectively attained the highest compressive strength at 90 days in Group 6. The maximum value achieved was 16MPa respectively.

Mix Design Reference	St	Strength at Days (MPa)			
	7	28	90	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - V-B5G20/RAF20/SSD60	4	8	10	0.15	2163
Mix 2 - RAF40/SSD60	2	5	16*	0.15	2379
Mix 3 - V-B5G20/RAF80	1	1	1	0.15	1989
Mix 4 - RAF60/SSD40	2	4	6	0.15	2301
Mix 5 - V-B5G15/RAF37.50/SSD45.50	4	8	12	0.15	2140
Mix 6 - RAF80/SSD20	2	3	3	0.15	2191
Mix 7 - V-B5G5/RAF47.50/SSD47.50	5	11	16*	0.15	2249
Mix 8 - V-B5G5/RAF67.50/SSD27.50	5	10	13	0.15	2142
Mix 9 - V-B5G10/RAF55/SSD35	4	9	10	0.15	2164
Mix 10 - V-B5G20/RAF50/SSD30	3	7	9	0.15	2065
Mix 11 - V-B5G10/RAF80/SSD10	2	4	6	0.15	2069
Mix 12 - V-B5G10/RAF30/SSD60	6	11	15	0.15	2321
Mix 13 - V-B5G15/RAF67.50/SSD17.50	3	4	9	0.15	2028
* Highlights highest compressive strength	in the group				

Table 6.18: Group 6 – Compressive Strength (V-B5G/RAF/SSD)

6.3.7.2 Contour Plots for 7, 28 and 90 Days Compressive Strength

The response data for the 7, 28 and 90 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.30, 6.31 and 6.32 respectively.



Figure 6.30: Group 6 – 7 Days Compressive Strength Contour Plot



Figure 6.31: Group 6 – 28 Days Compressive Strength Contour Plot



Figure 6.32: Group 6 – 90 Days Compressive Strength Contour Plot

6.3.7.3 Comparisons of 7, 28 and 90 Days Compressive Strength

Comparisons of the test results obtained from the 7, 28 and 90 days compressive strength are shown in Figures 6.33. All the mixes in this composition showed an increase in strength development as the specimen ages except for Mix 3 which showed a decrease in strength from 28 days to 90 days.



Figure 6.33: Comparisons of 7, 28 and 90 Days Compressive Strength

6.3.8 Group 7 Mixture Design Results

6.3.8.1 Mix Proportions and Compressive Strengths

The results for the 28, 90 and 180 days compressive strength tests for the V-B5G/SSD/BPD-C binder mixes are shown in Table 6.19. Mix 12, with the proportions of 20% V-B5G and 80% SSD attained the highest compressive strength at 28 and 90 days in Group 7. The maximum values achieved were 16MPa and 18MPa respectively.

Table 6.19: Group 7 – Compressive Strength (V-B5G/SSD/BPD-C)

Mix Design Reference	St	rength at Days (N			
	28	90	180	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - V-B5G36.25/SSD56.25/BPD-C7.5	5	9	9	0.15	2037
Mix 2 - V-B5G20.63/SSD68.13/BPD-C11.24	5	6	7	0.15	2074
Mix 3 - V-B5G32.50/SSD52.50/BPD-C15	5	6	9	0.15	1980
Mix 4 - V-B5G28.13/SSD68.13/BPD-C3.75	8	9	11	0.15	2139
Mix 5 - V-B5G5/SSD80/BPD-C15	7	15	16	0.15	2276
Mix 6 - V-B5G48.13/SSD48.13/BPD-C3.75	5	9	10	0.15	1967
Mix 7 - V-B5G48.13/SSD40.63/BPD-C11.25	5	7	7	0.15	1878
Mix 8 - V-B5G60/SSD25/BPD-C15	3	5	5	0.15	1808
Mix 9 - V-B5G60/SSD32.50/BPD-C7.5	4	5	6	0.15	1992
Mix 10 - V-B5G60/SSD40	6	8	9	0.15	2010
Mix 11 - V-B5G40/SSD60	8	11	12	0.15	2238
Mix 12 - V-B5G20/SSD80	16	18	18*	0.15	2606
Mix 13 - V-B5G12.5/SSD80/BPD-C7.5	10	14	16	0.15	2470
* Highlights highest compressive strength in	the group				
6.3.8.2 Contour Plots for 28, 90 and 180 Days Compressive Strength

Group 7 - Contour Plot

The response data for the 28, 90 and 180 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.34, 6.35 and 6.36 respectively.



Figure 6.34: Group 7 – 28 Days Compressive Strength Contour Plot



Figure 6.35: Group 7 – 90 Days Compressive Strength Contour Plot

118



Figure 6.36: Group 7 – 180 Days Compressive Strength Contour Plot

6.3.8.3 Comparisons of 28, 90 and 180 Days Compressive Strength

Comparisons of the test results obtained from the 28, 90 and 180 days compressive strength are shown in Figure 6.37. All the mixes in this composition showed an increase in strength development as the specimen.



Figure 6.37: Comparisons of 28, 90 and 180 Days Compressive Strength

6.3.9 Group 8 Mixture Design Results

6.3.9.1 Mix Proportions and Compressive Strengths

The results for the 7, 28 and 90 days compressive strength tests for the V-B5G/SSD/BPD-H binder mixes are shown in Table 6.20. Mix 13, with the proportions of 12.5% V-B5G, 80% SSD and 7.5% BPD-H attained the highest compressive strength at 28 and 90 days in Group 8. The maximum values achieved were 17MPa and 25MPa respectively.

Table 6.20: Group 8 – Compressive Strength (V-B5G/SSD/BPD-H)

Min Design Deference	Str	ength at [Days (MPa)	L/S	Dry Density
Mix Design Reference	7	28	90	Ratio	(Kg/m3)
Mix 1 - V-B5G36.25/SSD56.25/BPD-H7.5	3	5	7	0.15	2043
Mix 2 - V-B5G20.63/SSD68.13/BPD-H11.24	6	11	15	0.15	2374
Mix 3 - V-B5G32.50/SSD52.50/BPD-H15	3	5	7	0.15	2169
Mix 4 - V-B5G28.13/SSD68.13/BPD-H3.75	4	8	9	0.15	2278
Mix 5 - V-B5G5/SSD80/BPD-H15	9	16	18	0.15	2686
Mix 6 - V-B5G48.13/SSD48.13/BPD-H3.75	2	4	4	0.15	2062
Mix 7 - V-B5G48.13/SSD40.63/BPD-H11.25	2	2	3	0.15	1991
Mix 8 - V-B5G60/SSD25/BPD-H15	1	1	2	0.15	1962
Mix 9 - V-B5G60/SSD32.50/BPD-H7.5	1	1	2	0.15	1800
Mix 10 - V-B5G60/SSD40	1	6	8	0.15	1849
Mix 11 - V-B5G40/SSD60	2	8	11	0.15	2112
Mix 12 - V-B5G20/SSD80	6	16	18	0.15	2347
Mix 13 - V-B5G12.5/SSD80/BPD-H7.5	8	17	25*	0.15	2566
* Highlights highest compressive strength in	the g	roup			

6.3.9.2 Contour Plots for 7, 28 and 90 Days Compressive Strength

The response data for the 7, 28 and 90 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.38, 6.39 and 6.40 respectively.



Figure 6.38: Group 8 – 7 Days Compressive Strength Contour Plot



Figure 6.39: Group 8 – 28 Days Compressive Strength Contour Plot



Figure 6.40: Group 8 – 90 Days Compressive Strength Contour Plot

6.3.9.3 Comparisons of 7, 28 and 90 Days Compressive Strength

Comparisons of the test results obtained from the 7, 28 and 90 days compressive strength are shown in Figure 6.41. All the mixes in this composition showed an increase in strength development.



Figure 6.41: Comparisons of 7, 28 and 90 Days Compressive Strength

6.3.10 Group 9 Mixture Design Results

6.3.10.1 Mix Proportions and Compressive Strengths

The results for the 28, 90 and 180 days compressive strength tests for the PG/QWD/GGBS binder mixes are shown in Table 6.21. Mix 13, with the proportions of 10% PG, 30% QWD and 60% GGBS attained the highest compressive strength at 28, 90 and 180 days in Group 9. The maximum values achieved were 30MPa, 38MPa and 41MPa respectively.

Min Design Defenses	Str	rength at Days (N		Dury Davasitas (Karland)	
Mix Design Reference	28	90	180	L/S Ratio	Dry Density (Kg/m3)
Mix 1 - PG20/QWD20/GGBS60	26	34	37	0.17	1989
Mix 2 - PG10/QWD80/GGBS10	2	11	14	0.17	2151
Mix 3 - PG10/QWD55/GGBS35	21	21	32	0.17	2139
Mix 4 - PG5/QWD47.50/GGBS47.50	22	32	40	0.17	2137
Mix 5 - PG20/QWD50/GGBS30	13	29	33	0.17	2098
Mix 6 - PG15/QWD37.50/GGBS47.50	24	30	33	0.17	2003
Mix 7 - PG20/QWD80	1	1	1	0.17	2099
Mix 8 - QWD80/GGBS20	2	10	17	0.17	2117
Mix 9 - PG15/QWD67.50/GGBS17.50	6	22	26	0.17	2129
Mix 10 - QWD40/GGBS60	10	17	21	0.17	2068
Mix 11 - QWD60/GGBS40	5	14	21	0.17	2168
Mix 12 - PG5/QWD67.50/GGBS27.50	18	31	35	0.17	2127
Mix 13 - PG10/QWD30/GGBS60	30	38	41*	0.17	1976
* Highlights highest compressive strength	in the group				

Table 6.21: Group 9 – Compressive Strength (PG/QWD/GGBS)

6.3.10.2 Contour Plots for 28, 90 and 180 Days Compressive Strength

The response data for the 28, 90 and 180 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.42, 6.43 and 6.44 respectively.



Figure 6.42: Group 9 – 28 Days Compressive Strength Contour Plot



Figure 6.43: Group 9 – 90 Days Compressive Strength Contour Plot



Figure 6.44: Group 9 – 180 Days Compressive Strength Contour Plot

6.3.10.3 Comparisons of 28, 90 and 180 Days Compressive Strength

Comparisons of the test results obtained from the 28, 90 and 180 days compressive strength are shown in Figure 6.45. All the mixes in this composition showed an increase in strength development as the specimen ages.



Figure 6.45: Comparisons of 28, 90 and 180 Days Compressive Strength

6.3.11 Group 9A Mixture Design Results

6.3.11.1 Mix Proportions and Compressive Strengths

The results for the 28 and 90 days compressive strength tests for the V-B5G/QWD/GGBS binder mixes are shown in Table 6.22. Mix 6, with the proportions of 15% V-B5G, 37.5% QWD and 47.5% GGBS attained the highest compressive strength at 28 days. The highest compressive strength at 90 days was Mix 4 with the proportions 5% V-B5G, 47.5% QWD and 47.5% GGBS. The maximum values achieved for the 28 and 90 day strengths were 26MPa and 35MPa respectively.

Mix Design Deference	Strength at	Days (MPa)	L/C Datia	Duy Donoity (Ka /m2)	
Mix Design Reference	28	90		Dry Density (Kg/ms)	
Mix 1 - V-B5G20/QWD20/GGBS60	20	30	0.19	2126	
Mix 2 - V-B5G10/QWD80/GGBS10	2	16	0.19	2261	
Mix 3 - V-B5G10/QWD55/GGBS35	17	33	0.19	2158	
Mix 4 - V-B5G5/QWD47.50/GGBS47.50	24	35*	0.19	2217	
Mix 5 - V-B5G20/QWD50/GGBS30	20	30	0.19	2113	
Mix 6 - V-B5G15/QWD37.50/GGBS47.50	26	34	0.19	2087	
Mix 7 - V-B5G20/QWD80	1	1	0.19	2236	
Mix 8 - QWD80/GGBS20	3	15	0.19	2393	
Mix 9 - V-B5G15/QWD67.50/GGBS17.50	4	18	0.19	2177	
Mix 10 - QWD40/GGBS60	11	19	0.19	2208	
Mix 11 - QWD60/GGBS40	10	24	0.19	2293	
Mix 12 - V-B5G5/QWD67.50/GGBS27.50	16	28	0.19	2300	
Mix 13 - V-B5G10/QWD30/GGBS60	21	31	0.19	2105	
* Highlights highest compressive strength in	n the group				

Table 6.22: Group 9A – Compressive Strength (V-B5G/QWD/GGBS)

6.3.11.2 Contour Plots for 28 and 90 Days Compressive Strength

The response data for the 28 and 90 days compressive strength was entered in Minitab 18 software programme and the results of the mixture contour plots were produced as shown in Figures 6.46 and 6.47 respectively.



Figure 6.46: Group 9A – 28 Days Compressive Strength Contour Plot



Group 9A - Contour Plot (90 Day Strength - V-B5G/QWD/GGBS)

Figure 6.47: Group 9A – 90 Days Compressive Strength Contour Plot

6.3.11.3 Comparisons of 28 and 90 Days Compressive Strength

Comparisons of the test results obtained from the 28 and 90 days compressive strength are shown in Figure 6.48. All the mixes in this composition showed an increase in strength development as the specimen ages. Mix 7 showed no change in strength development from 28 days to 90 days.



Figure 6.48: Comparisons of 28 and 90 Days Compressive Strength

6.3.12 Binary Group - PG/GGBS

Table 6.23 shows the binary compositions of PG/GGBS. The values for the binary compositions were obtained from Group 1 (PG/MHT/GGBS), 3 (PG/GGBS/BPD-C) and 4 (PG/GGBS/BPD-H) of this research study. At 7 and 28 days, the highest compressive strength showed in Figure 6.49 was obtained from the proportions of 20% PG and 80% GGBS. The maximum value achieved in the binary group at 28 days was 19MPa. There was an increase in strength gain when the percentage of GGBS increases 20% by weight. In other words, higher content of PG in the mix, resulted in lower strength development in the paste mixtures.

Mix Design Deference	Strength at	: Days (MPa)	L/C Datia	Dry Density (Kg/m3)					
wix Design Reference	7	28							
PG20/GGBS80	3	19*	0.15	1755					
PG40/GGBS60	3	16	0.15	1943					
PG60/GGBS40	2	8	0.15	1842					
PG80/GGBS20	3	3	0.15	1865					
* Highlights highest compressive strength in the group									

Table 6.23: Binary Group – Compressive Strength (PG/GGBS)



Binary Group - PG/GGBS (Strength in MPa)

Figure 6.49: Binary Group - Comparisons of 7 and 28 Days Compressive Strength

6.4 Phase Two - Validation of Prediction for chosen Groups

6.4.1 Understanding Coefficient Equation

The coefficient estimate used to calculate the predicted values of the preferred mixes is derived from Equation 1 (Eq. 1)

$$Y_{ijk} = \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 \quad \text{[Equation 6.1]}$$

Where Y_{ijk} represent the response for the *i*th proportion of the component 1 (X_1), the *j*th proportion of component 2 (X_2) and the k^{th} proportion of component 3 (X_3). Therefore, using Group 1 with the components PG, MHT and GGBS as an example:

 $Y_{PG,MHT,GGBS}$ represent the response when $(X_1) = PG$, $(X_2) = MHT$ and $(X_3) = GGBS$. X_1 , X_2 and X_3 are the variables and represent the response in each mix design and β_1 , β_2 and β_3 are the coefficient of the terms which are constant.

6.4.2 Group 2 Validation of Prediction

Table 6.24 illustrates the predicted and actual compressive strength results at 28 days for Group 2. The predicted values derived from the Coefficient Equation – Eq. 2.

$$CS = -821.884(PG)-11.9009(RAF)-11.2269(GGBS)+1059.19$$

(PG*RAF)+1203.40(PG*GGBS)+79.2780(RAF*GGBS) [Equation 6.2]

Even though the percentage error range varies, the average percentage error for the top exceeding 20MPa in Group 2 was 8.5%. Mix 4 (Vfour mixes B5G5/RAF47.50/GGBS47.50), the highest compressive strength (27MPa) had 28% error. The difference in percentage error in the other mixes in the group could have been due to a number of factors. Comparisons of the predicted and actual compressive strength for Group 2 at 28 days strength are shown in Figure 6.50.

Mix	PG (%)	RAF (%)	GGBS (%)	Predict 28 Days comp. strength (MPa)	Actual 28 Days comp. strength (MPa)	Error %
Mix 1	20.00	20.00	60.00	22.8	23.7	4
Mix 2	10.00	80.00	10.00	10.3	2.3	353
Mix 3	10.00	55.00	35.00	23.0	23.7	3
Mix 4	5.00	47.50	47.50	19.5	27.0	28
Mix 5	20.00	50.00	30.00	16.3	14.3	14
Mix 6	15.00	37.50	47.50	26.4	24.7	7
Mix 7	20.00	80.00	0.00	-4.4	0.8	647
Mix 8	0.00	80.00	20.00	0.9	2.6	64
Mix 9	15.00	67.50	17.50	14.9	10.6	40
Mix 10	0.00	40.00	60.00	7.5	3.9	95
Mix 11	0.00	60.00	40.00	7.4	2.8	160
Mix 12	5.00	67.50	27.50	14.8	22.8	35
Mix 13	10.00	30.00	60.00	25.8	25.8	0

Table 6.24: Group 2 – Predicted and actual compressive strength at 28 days



Figure 6.50: Group 2 – Predicted and actual compressive strength at 28 days

6.4.3 Group 2A Validation of Prediction

Table 6.25 illustrates the predicted and actual compressive strength results at 28 days for Group 2A. The predicted values derived from the Coefficient Equation – Eq. 3.

CS = -753.639(V-B5G)-17.2196(RAF)-251896(GGBS)+1006.09 (V-B5G*RAF)+1175.33(V-B5G*GGBS)+117.031(RAF*GGBS) [Equation 6.3]

Even though the percentage error range varies, the average percentage error for the top four mixes exceeding 25MPa in Group 2A was 3.25%. Mix 6 (V-B5G15/RAF37.50/GGBS47.50), the highest compressive strength (29.45MPa) had only 1% error. The difference in percentage error in the other mixes in the group could have been due to a number of factors. Comparisons of the predicted and actual compressive strength for Group 2A at 28 days strength are shown in Figure 6.51.

Mix	V-B5G (%)	RAF (%)	GGBS (%)	Predict 28 Days comp. strength (MPa)	Actual 28 Days comp. strength (MPa)	Error %
Mix 1	20.00	20.00	60.00	26.0	25.2	3
Mix 2	10.00	80.00	10.00	9.9	3.5	183
Mix 3	10.00	55.00	35.00	25.4	26.2	3
Mix 4	5.00	47.50	47.50	20.4	24.1	15
Mix 5	20.00	50.00	30.00	21.8	21.7	1
Mix 6	15.00	37.50	47.50	29.7	29.4	1
Mix 7	20.00	80.00	0.00	-3.5	0.5	884
Mix 8	0.00	80.00	20.00	-0.1	2.6	104
Mix 9	15.00	67.50	17.50	17.5	12.9	35
Mix 10	0.00	50.00	60.00	6.1	3.9	57
Mix 11	0.00	60.00	40.00	7.7	2.8	170
Mix 12	5.00	67.50	27.50	15.6	22.0	29
Mix 13	10.00	30.00	60.00	26.1	27.8	6

Table 6.25: Group 2A – Predicted and actual compressive strength at 28 days



Figure 6.51: Group 2A – Predicted and actual compressive strength at 28 days

6.4.4 Group 9 Validation of Prediction

Table 6.26 illustrates the predicted and actual compressive strength results at 28 days for Group 9. The predicted values derived from the Coefficient Equation – Eq. 4.

$$CS = -633.958(PG) - 7.30408(QWD) + 18.8068(GGBS) + 807.383$$

(PG*QWD)+911.116(PG*GGBS)+19.1738(QWD*GGBS) [Equation 6.4]

Even though the percentage error range varies, the average percentage error for the top four mixes exceeding 25MPa in Group 9 was 3.75%. Mix 13 (PG10/QWD30/GGBS60), the highest compressive strength (30.2MPa) had 7% error. The difference in percentage error in the other mixes in the group could have been due to a number of factors. Comparisons of the predicted and actual compressive strength for Group 9 at 28 days strength are shown in Figure 6.52.

Mix	PG (%)	RAF (%)	GGBS (%) Predict 28 Days comp. strength (MPa) A		Actual 28 Days comp. strength (MPa)	Error %
Mix 1	20.00	20.00	60.00	27	26.5	2
Mix 2	10.00	80.00	10.00	7.9	2.2	253
Mix 3	10.00	55.00	35.00	19.2	20.7	7
Mix 4	5.00	47.50	47.50	18.9	21.6	13
Mix 5	20.00	50.00	30.00	13.5	13.1	3
Mix 6	15.00	37.50	47.50	24.8	24.2	3
Mix 7	20.00	80.00	0.00	-3.5	0.9	497
Mix 8	0.00	80.00	20.00	1	2.3	57
Mix 9	15.00	67.50	17.50	11.2	5.6	100
Mix 10	0.00	40.00	60.00	13	9.8	33
Mix 11	0.00	60.00	40.00	7.7	5.5	41
Mix 12	5.00	67.50	27.50	11.9	18.1	34
Mix 13	10.00	30.00	60.00	28	30.2	7

Table 6.26: Group 9 – Predicted and actual compressive strength at 28 days



Figure 6.52: Group 9 – Predicted and actual compressive strength at 28 days

6.5 Phase Two - Refining the chosen Groups

The compressive strength results for the mixes in Group 2 (PG/RAF/GGBS), 2A (V-B5G/RAF/GGBS) and 9 (PG/QWD/GGBS) showed that they have potential and have exceeded expectation in regards the objectives of this research study. Mix 4 (PG5/RAF47.50/GGBS47.50) in Group 2, Mix 6(V-B5G15/RAF37.50/GGBS47.50) in Group 2A and Mix 13 (PG10/QWD30/GGBS60) in Group 9 had compressive strengths of up to 27 MPa, 29 MPa and 30 MPa respectively at 28 days and, have been identified as the mixtures with the highest compressive strengths in the phase one experiment.

As the results of Groups 2, 2A and 9 were considered to have potential, further laboratory tests were carried out to determine their optimum performance. All three groups were refined to establish whether the 15% liquid / solid ratio used in the initial experiment was the optimum water content required to achieve its full potential. In addition to the 15% ratio used in the initial experiment, a liquid / solid ratio of 13, 17, 19 and 21% was used to determine their optimum performance. The specimens of the 13 and 17% ratios were tested at 14, 28 and 90 days for Group 2. Specimens for the 17, 19 and 21% ratios were tested at 28 and 90 days for Group 2A and specimens for the 13, 17, 19 and 21% ratios were tested at 7, 28 and 90 days for Group 9. Unlike the tests (7 and 28 days) carried out on the 15% ratio in the initial experiment, the 14 and 90 days tests were to better understand the performance with regard to early and long-term strength of the mixtures and, the effect on compaction and hydration. Comparisons of the optimum water contents of the three preferred groups at 28 days strength are shown in Figure 6.53. The compressive strength results comparing the 13, 15 and 17% ratio at 28 days for Group 2 are shown in Table 6.27.



Figure 6.53: Group 2, 2A and 9 – Comparisons of liquid / solid ratios at 28 days strength

Table 6.27: Group 2 – Comparisons of 13,	15 and 17% liquid / solid ratio
compressive strength results at 28 days	

Mix Design	Proport	tions	Compressive Strength (MPa)									
Reference	PG	RAF	CCBS	13% L/S Ratio			15% L/	15% L/S Ratio		17% L/S Ratio		
	-		0000	14 Days	28 Days	90 Days	7 Days	28 Days	14 Days	28 Days	90 Days	
Mix 1	20	20	60	2	13	26	2	24	13	21	41	
Mix 2	10	80	10	2	3	16	1	2	0	2	10	
Mix 3	10	55	35	2	13	34	1	24	4	16	28	
Mix 4	5	47.50	47.50	4	12	26	1	27*	9	19	30	
Mix 5	20	50	30	2	7	19	1	14	3	15	34	
Mix 6	15	37.50	47.50	3	13	25	1	25	9	18	35	
Mix 7	20	80	0	1	1	1	1	1	1	1	1	
Mix 8	0	80	20	2	5	15	1	3	2	7	15	
Mix 9	15	67.50	17.50	2	4	22	1	11	1	4	16	
Mix 10	0	40	60	3	6	12	1	4	2	7	14	
Mix 11	0	60	40	2	6	14	1	3	2	6	14	
Mix 12	5	67.50	27.50	3	12	25	2	23	2	18	26	
Mix 13	10	30	60	8	16*	26	1	26	18	25*	37	
* Highlights hi	ghest c	ompres	sive stre	ength in 13	8, 15 and 1	7% liquid/	solid ratio	at 28 days	6			

CHAPTER 7 – PHASE ONE AND TWO ANALYSIS AND DISCUSSIONS

7.1 Introduction

This chapter evaluates the hydration and pozzolanic effects on the chosen groups. Evaluations of the liquid / solid ratios for some of the groups were explored and decisions made on the optimum water content used in the experiments. The short and long-term behaviour of the chosen group with regards to compressive strength are also discussed in this chapter.

7.2 Evaluation of Hydration and Pozzolanic Effects on the Chosen Groups

Laboratory experiments or investigations were not carried out on the specimens in this research study to evaluate hydration and possible pozzolanic effects of the chosen groups discussed in this section. It was assumed that hydration in the raw materials determined during the XRD experiments remained unchanged, therefore this information was used as a baseline to evaluate the hydration of the specimens derived from dry hydraulic cement pastes.

The hydration process is a key element that determines the early and long-term performance of the cement paste. The physical behaviour of cement during hydration is similar to tricalcium silicate (C_3S) and dicalcium silicate (C_2S) compounds alone. These two calcium silicates are the two main cementitious compounds in cement, which together comprise 75 – 85 of Portland cements and contribute heavily to binding and strength.

On the mass basis, both silicates require approximately the same amount of water for their hydration. C_3S produces more than twice as much calcium hydroxide ($Ca(OH)_2$) as is formed by the hydration of C_2S . When hydration takes place, C_3S is believed to undergo hydrolysis producing calcium silicate hydrate ($C_3S_2H_3$), with the released lime separating out as $Ca(OH)_2$ (Neville 2012).

According to (Eglinton 1987), C_2S hydrates much more slowly. The greater part of its hydration does not take place until after about 28 days, but it continues beyond one year. C_3S on the other hand contributes to the strength of the cement paste at later age. The

slow rate of hydration means that the heat of hydration is low. The hydration reaction of both C_3S and C_2S is the formation of a gel of calcium silicate hydrate (C-S-H), which is the main binding agent in the cement paste, and of crystalline calcium hydroxide (Ca(OH)₂).

According to (Mehta and Monteiro 2017), C-S-H makes up 50 to 60 percent of the volume of solids in a completely hydrated Portland cement paste and provides the majority of the long-term strength and durability. It is known as the most important phase in determining the properties of the paste. $Ca(OH)_2$ on the other hand constitutes 20 to 25 percent of the volume of solids in the hydrated paste. However, Neville 2012 stated that C-S-H gel formed by pozzolanic activity incorporates a certain amount of alkalis and thus lowers the value of pH.

It was noticeable from the results of the compressive strength tests obtained from this research study that the groups containing pozzolans exhibit slower strength development. Figure 7.5 mentioned in Section 7.6 of this report shows rapid strength development from 7 to 28 days. Mehta et al. 2017 stated that when blends containing pozzolans are adequately reactive and are used in moderate proportions, and cured for long periods under moisture, the strength will increase. According to (Mehta and Monteiro 2006), pozzolanic admixtures such as fly ash reacts with calcium hydroxide to form a reaction product that is similar in composition and properties to C-S-H. The pozzolanic reaction is also accompanied by a reduction in the total volume and size of capillary pores – an effect that is equally important for the enhancement of strength.

(Liu, Xie and Li 2005), stated that the use of Portland cement with pozzolans generate a reaction between calcium hydroxide produced from Portland cement hydration and the amorphous SiO₂ of the glass phase from pozzolan materials. This results to an increase in the calcium silicate hydrate (C-S-H) gel, thus increasing density and strength.

(Eglinton 1987), stated that the inclusion of pozzolana in a mixture increases the impermeability of the concrete paste, rendering it less vulnerable to ingress of aggressive solutions; that as the concentration of $Ca(OH)_2$ in the pore water is reduced the calcium aluminates become more soluble and ettringite is formed in an aqueous, not solid, phase and that because of the increased amount of silica contributed by the pozzolan.

Based on the findings of the study carried out by (Abdullah et al. 2012), the strength development behaviour of Groups 2, 2A, and 9 of this research study is a typical example of pozzolan. Its slower strength development at early age of 7 / 14 days and, longer setting time to achieve its optimum strength at 28 / 90 days exhibits the strength development behaviour of pozzolans.

7.3 The Effect of Different Liquid / Solid Ratios on Short to Medium-term Compressive Strength

The hydration process is one of the key elements that determines the short and long-term performance of the hydraulic paste mixtures in terms of strength development. It was very noticeable at 28 days strength using 13, 15 and 17% liquid / solid ratios that 100% hydration was achieved with the mixtures of 15% ratio. There was sharp decrease in strength at 13% compared to a moderate decrease at 17%. One of the main characteristics on the effect of workability on the semi-dry hydraulic paste mixture was compaction of the sample. (Jackson and Dhir 1996), defined compactability as the ease a mix can be fully compacted to remove all trapped air. There is no doubt that the 15% water content achieved full compaction and optimum mechanical performance at 28 days of the mixtures in the Group. Figure 7.1 illustrates the highest compressive strength on Group 2 with different water content.



Figure 7.1: Group 2 – Comparison of replacement of 13, 15 and 17% liquid / solid ratio

It can also be said that the low strength results with mixtures of 13% ratio was the effect on compaction and not enough water available to carry on during curing time. It was also observed that the mixtures of 17% ratio was the second strongest in terms of compressive strength achieved at 28 days when compared to the other ratios used in refining Group 2 (PG/RAF/GGBS). The not so good results for the 17% ratio compared to the 15% ratio may have been as a result of less workability in excess water, that evaporates and leaves pores thereby reducing the strength. It clearly shows that the mixtures of 17% ratio had more than required water that was needed.

Comparisons of the test results obtained at 28 days compressive strength are shown in Figure 7.2.



Figure 7.2: Comparisons of 13, 15 and 17% liquid / solid ratio at 28 days Compressive Strength

It can be said that there is a possibility that the curing temperature has had an effect on hydration on the mixtures with 13 and 17% liquid / solid ratio. A study on binders alternative to Portland cement carried out by (Juenger et al. 2011) whereby the curing temperature during hydration was found to affect the strength of the cement paste. They mentioned in their report that at low curing temperatures, metastable hydrates CAH_{10} and C_2AH_8 form.

The predominant metastable hydrate formed at temperatures below 15° C, CAH₁₀ is also formed and this is generally accepted. C₂AH₈ is formed as the curing temperature increases to 30° C. The conversion of these hydrates to the stable C₃AH₆ phase is accompanied by the formation of AH₃ gel and the release of water known as thermodynamically inevitable process. The paste increases in porosity and the strength of the material decreases as a result of the conversion process.

Another factor that may have influenced the mechanical performance of Groups 2, 2A and 9 was the physical properties of the materials in the components. Analysis showed that the average particle size distribution of the materials (PG, V-B5G, RAF, QWD and GGBS) used in the preferred groups with the highest compressive strengths is in the region of approximately 0.098 mm diameter (under 100 micron). (Ganjian, Eshmaiel, Claisse and Sadeghi-Pouya 2007) report that pozzolanic materials with a finer particle size results in faster hydration and reduced setting time of the binder. This is due to the higher surface area and electric charges induced on the surface of the particles during the grinding process.

The author believes that the dominant pozzolans in the three groups with the optimum mixes may have been a contributory factor to their success. It is also very likely that the sharp increase in strength development may have been as the results of high levels of alkalis. High alkalinity in the constituents of PG/RAF/GGBS, V-B5G/RAF/GGBS and PG/QWD/GGBS may have assured hydraulically active mixes.

7.4 The Effect of Different Liquid / Solid Ratios on Long-term Compressive Strength

Group 2 (PG/RAF/GGBS) was used to evaluate the effect of longer period of curing. The development of compressive strength of selected Mix 1 (PG20/RAF20/GGBS60), Mix 4 (PG5/RAF47.50/GGBS47.50), Mix 6 (PG5/RAF37.50/GGBS47.50) and Mix 13 (PG10/RAF30/GGBS60) at 7, 14, 28 and 90 days using liquid / solid ratios of 13, 15 and 17% were evaluated.

Unlike the compaction achieved with the mixtures of 15% liquid / solid ratio at 28 days as discussed in Section 7.3 of this study, at 90 days there was a change in liquid / solid ratio percentage in the top three mixes with the highest compressive strength. The trend of the

long-term strength results in Mixes 1 (PG20/RAF20/GGBS60), 6 (PG5/RAF37.50/GGBS47.50) and 13 (PG10/RAF30/GGBS60) was different to that of Mix 4 (PG5/RAF47.50/GGBS47.50). Figure 7.3 shows the development of compressive strength of the selected mixes using liquid / solid ratio of 13, 15 and 17%.

In Mix 4 (PG5/RAF47.50/GGBS47.50), long-term strength development in the different liquid / solid ratios (13, 15 and 17%) increase as the specimens ages with no interchange between the different liquid / solid ratios. In Mix 1 (PG20/RAF20/GGBS60), Mix 6 (PG5/RAF37.50/GGBS47.50) and Mix 13 (PG10/RAF30/GGBS60), there was a change in strength development between 15 and 17% ratio as the specimens ages from 28 days to 90 days. There is no doubt the longer period curing with 17% water content achieved full compaction and optimum mechanical performance. The change in mechanical performance for the 17% ratio at 90 days on Mixes 1, 6 and 13 compared to Mix 4 that performed like conventional cement where an interchange in strength development is not expected as the specimen ages. The difference in mechanical behaviour of Mixes 1, 6 and 13 to that of Mix 4 may have been the results of the following:

- i. reactivity of the greater percentage of the GGBS in Mixes 1, 6 and 13 may have been one of the factors that influenced the long-term strength development. The constituents of the GGBS in Mixes 1, 6 and 13 was 60, 47.50 and 60% respectively and therefore a dominant factor. Unlike Mix 4, there was an equal amount of GGBS to that of RAF (47.50% each). This equal proportion of GGBS and RAF in Mix 4 may have explained the reason why there was no interchange between the liquid / solid ratio with regards to the long-term compressive strength between 28 days and 90 days when compared to the other three mixes; and
- ii. variations in the water content is another factor that may have influenced the longterm compressive strength of the selected mixes. Initial hydration is known to be slow as shown in Figure 7.3 below and explained in Section 7.3 when 15% water content achieved full optimum mechanical performance at 28 days. At 90 days with 17% water content, the ultimate strengths of the mixes were achieved.

Based on the analysis of the selected mixes, it can be said that the long-term strength development may have been attributed to not only the 17% water content but, also the



effect of the greater percentage of GGBS in the mixes.

Figure 7.3: Development of compressive strength on selected mixes using liquid / solid ratio of 13, 15 and 17% at 7, 14, 28 and 90 days

7.5 The Effect of PG and V-B5G in Mixes Containing RAF and GGBS

Group 2 and 2A had mixes containing PG/RAF/GGBS and V-B5G/RAF/GGBS respectively. Selected Mixes 1, 4, 6 and 13 with the same proportions of the different materials in the two Groups (2 and 2A) have been used to evaluate the effect of PG and V-B5G in mixes containing RAF and GGBS at 7, 14, 28 and 90 days. The effect of PG and V-B5G have been based on the optimum liquid / solid ratios at 90 days to achieve the strongest mechanical performance of the mixes. The optimum ratio of 17% was used for the PG/RAF/GGBS combination and 19% for the V-B5G/RAF/GGBS. Figure 7.4 shows

comparison of PG and V-B5G in the selected mixes containing RAF and GGBS.

Analysis of the results in general showed that there was not a great effect of PG and V-B5G in mixes containing RAF and GGBS. The selected mixes on the two groups performed similarly in their short and long-term strength development. For the mediumterm (14 - 28 days) strength, all the mixes performed similarly except Mix 4 with the average strength difference of approximately 10MPa. At 7 days, the mixes on Group 2A had high compressive strength results when compared to Group 2, except for Mix 4 which was marginal. The 90 days strength on the other hand showed that Mixes 4, 6 and 13 had similar strength development except for Mix 1 where the strength difference was approximately 8MPa.



Figure 7.4: Comparisons on the effects of PG and V-B5G on selected mixes containing RAF and GGBS

7.6 The Effect of RAF and QWD in Mixes Containing PG and GGBS

Group 2 and 9 have mixes containing PG/RAF/GGBS and PG/QWD/GGBS respectively. Selected Mixes 1, 4, 6 and 13 with the same proportions of the different materials in the two Groups (2 and 9) have been used to evaluate the effect of RAF and QWD in mixes containing PG and GGBS at 7, 28 and 90 days. The effect of RAF and QWD have been based on the optimum water contents at 90 days to achieve the strongest mechanical performance of the mixes. The optimum water content of 17% was used for the PG/RAF/GGBS and PG/QWD/GGBS combinations. Figure 7.5 shows comparison of RAF and QWD in the selected mixes containing PG and GGBS.

Analysis of the results show that the presence of QWD when compared to RAF in the pastes containing PG and GGBS does not cause a decrease in short, medium and longterm strength development. In general, both the QWD and RAF in the two different groups behaved similarly with regard to strength development. However, it was recognised that there was a notable difference in the early strength development on some of the selected mixes with QWD. The percentage by mass of the major chemical oxides (SiO₂ + Fe₂O₃ + Al₂O₃) for both the RAF and QWD are similar. The main difference between the RAF and QWD is that one is a natural pozzolan and the other is an artificial pozzolan (also referred to as natural calcined pozzolan). Both materials are pozzolans and conform with requirements set out in (ASTM 2013), whereby the activity index is over 75% of the controlled mix at 28 days. They are also in line with the requirements set out in (EN, BS 2011) whereby the materials consist predominantly of reactive silicon dioxide (SiO₂) and aluminium oxide (Al₂O₃). The content of the reactive silicon dioxide (SiO₂) shall be not less than 25% by mass. (Astm 2003) stipulates that for a material to be pozzolanic, it should meet the requirement content of pozzolanic oxides (SiO₂ + Fe₂O₃ + Al₂O₃) of more than 65 – 70%. The XRF analysis of the RAF carried out on two separate batches (2017 and 2018) showed the samples to be approximately 65 and 59% respectively. On the other hand, analysis on the QWD showed the pozzolanic oxides content to be approximately 60%. As the percentage by mass of the major oxides are similar in both the RAF and QWD, the decrease in the short-term strength development in mixes containing RAF may have been the results of both mineralogical properties and / or the minor chemical oxides





Figure 7.5: Comparisons on the effects of RAF and QWD on selected mixes containing PG and GGBS

7.7 Strength Development of Hydraulic Paste

There was a rapid increase in strength development between the 7 and 28 days in all the preferred groups (2 PG/RAF/GGBS), (2A V-B5G/RAF/GGBS) and (9 PG/QWD/GGBS). This rapid increase may have been attributed to the presence of RAF, QWD and GGBS in the mixes. Figure 7.6 shows an increase in strength development at 14, 28 and 90 days on Group 2 with liquid / solid ratio of 17% as the specimen ages.

In general, looking at the trends of this research study's findings, one can hypothesise that the short and long-term strength development of the hydraulic paste containing GGBS are in agreement with reports carried out by (Hogan and Meusel 1981; Roy 1982). Their

research found that the strength development was slow during the early stages of hydration for concrete containing 40 – 60% GGBS as cement replacement. In a separate study carried out by (Khatib and Hibbert 2005), they reported similar findings that the presence of GGBS is highly beneficial between 40 and 60% replacement. They acknowledged a reduced strength during the first 28 days. After 28 days and up to 90 days, they concluded the strength increases with the presence of GGBS of up to 60%. A similar review carried out by (Suresh and Nagaraju 2015) confirmed that a GGBS concrete will have lower strength at early ages but its long-term strength will be greater, the reduction in the early strength development is generally noticeable at higher GGBS levels and low temperatures. (Aldea et al. 2000; Hwang and Lin 1986; Miura and Iwaki 2000) reported that the benefit in strength of concrete containing 20 – 60% GGBS does not occur until after 28 days of curing, where similar or higher long-term strength was obtained as compared with that of normal PC concrete. A study carried out by (Siddique 2014) on utilization of iron and steel industry by-product (GGBS) in concrete concluded that the use of GGBS does not only accelerate the hydration process, an amount between 40 - 60% increases the compressive strength, split tensile strength and flexural strength of concrete.



Figure 7.6: Development of compressive strength as the specimens age with liquid / solid ratio of 17%

Generally, industrial by-products including those used in this research study are not conventional materials and therefore not expected to behave exactly like traditional materials. Industrial by-products of this sort are highly acceptable for their ultimate strength (80- 90%) to be achieved at 90 days of curing. Figure 7.7(a) & (b) shows the percentage of strength development of Group 2 Mix 1 (PG20/RAF20/GGBS60) at 3, 7, 14, 28 and 90 days as the specimen ages.



Figure 7.7(a) & (b): Percentage of Strength Development

Given the novelty of this research study and the introduction of new industrial by-products to fully replace cement, a direct comparison of the strength gained as the specimen ages on the preferred mixtures cannot be made to similar materials as most research concentrates on partial cement replacement. However, a review carried out by (Suresh and Nagaraju 2015) concluded that a 50% by weight of GGBS concrete will typically achieve approximately 45 to 55% of its 28 days strength at seven days, with a gain of about 20% from 28 to 90 days.

7.8 Discussions

There are a few key factors that may have influenced the strength of the preferred paste mixes on Groups 2 (PG5/RAF47.50/GGBS47.50), 2A (V-B5G15/RAF37.50/GGBS47.50) and 9 (PG10/QWD30/GGBS60). They are summarised as follows:

- the chemical composition of the materials;
- the mineralogical properties of the materials;

- the constituents of the mix itself with regards to the proportions of the different materials present. Ensuring that the correct boundaries of components in the design stage are met;
- the correct percentage of liquid / solid ratios used in the mixture with regards to the determination of optimum water in the groups; and
- the effect on hydration process during curing.

In addition to some of the key factors that may have influenced the mechanical performance of the hydraulic pastes mentioned above, there are other factors that may affect the outcome of the test results, and these are summarised as follows:

- using of the correct proportions of the different binary and ternary samples;
- ensuring consistency in mixing time of the samples;
- ensuring the correct amounts of samples in each mould and sequence in placing of the samples;
- ensuring correct and secured equipment is used for curing the specimens;
- ensuring the correct face and orientation of the specimens are placed on the surface areas of the compressive strength machine;
- avoid debris on surface areas of the lower and upper plate of the compressive strength machine; and
- ensuring the consistency in load application during testing is maintained.

It can be said that the dominant factor of calcium sulfate - $CaSO_4$ (CaO + SO₃) in the V-B5G material, calcium sulfate dihydrate - ($CaSO_42H_2O$) in the PG material, calcium silicate - $CaSiO_3$ (CaO + SiO₂) in the GGBS and the pozzolanic activity (SiO₂ + Fe₂O₃ + Al₂O₃) present in the RAF heavily influenced the performance of the concrete at both early and late age strengths.

It is also highly likely that the mineralogical properties of the PG / V-B5G may have had a great influence on the strength development of the optimum mixes. (Claisse, PA and

Ganjian 2006), distinguished the three main compounds of calcium sulphate and are summarised as follows:

- i. anhydrite (CaSO₄) compound with no water molecules;
- ii. hemihydrate $(CaSO_4 0.5H_20)$ compound with partial water; and
- iii. dihydrate (CaSO₄ 2H₂0) compound fully hydrated with water and correctly described as gypsum.

The peaks and valleys pattern obtained from the X-ray diffraction (XRD) analysis of the V-B5G showed the presence of high levels of gypsum (CaSO₄ – $2H_20$) and a moderate level of basanite (CaSO₄ – $0.5H_20$) also referred to hemihydrate. One of the two main industrial uses of these compounds mentioned by (Claisse, PA and Ganjian 2006) is in cement. Cement producers use a blend of gypsum and anhydrite as a set controller.

The GGBS used in this research study meets the requirements set out in (EN, BS 2011) for use in blended cement. At least two-thirds of the total mass of slag must consist of the sum of CaO, MgO and SiO₂. Also, the ratio of the mass of CaO plus MgO to that of SiO₂ must exceed 1.0. The major oxides present in the GGBS used in this research study were calcium (CaO), aluminate (Al₂O₃) and silicate (SiO₂). These three oxides total over 85 percent of the oxide composition present in the sample. The sum of CaO, MgO and SiO₂ of the total mass of slag was more than two-thirds and the ratio of the mass of CaO plus MgO to that of SiO₂ the total mass of slag was more than two-thirds and the ratio of the mass of CaO plus MgO to that of SiO₂ exceeds 1.0. This did not only show that the material obtained from Hanson Hiedel-berg Cement Group was compliant with (EN, BS 2011) and suitable for use as blended cement, it also shows that the material had a chemical composition that conforms to the criteria for common cements.

CHAPTER 8 – PHASE THREE EXPERIMENTAL TESTS AND CHOOSING THE OPTIMUM BLEND

8.1 Introduction

This chapter covers the results of further experimental tests carried out on the three top preferred mixes mentioned in Chapters 6 and 7 to determine the optimum mix known as the 'Warwickshire Blend.' Tests to determine the long-term durability of the three mixes were used to distinguish which of the mixes regarded as the optimum blend, as optimisation is not about the mix with the highest compressive strength, it is ensuring that a defendable margin is achieved due to the very close nature of the mechanical performance of the three preferred mixes discovered in previous chapters.

In addition to the compressive strength test results obtained from the top three mixes identified to have potential on Groups 2 (Mix 1 - PG20/RAF20/GGBS60), 2A (Mix 13 - VB5G10/RAF30/GGBS60) and 9 (Mix 13 - PG10/QWD30/GGBS60), frost susceptibility and high-pressure permeability tests were carried out to determine the Warwickshire novel blend.

8.2 Frost Susceptibility – Resistance to Freezing and Thawing

Laboratory tests to determine resistance to freezing and thawing of the hydraulically bound mixtures were carried out in accordance with (CEN/TS). The freeze-thaw cycle of the three mixes (Mix 1 - Group 2, Mix 13 – Group 2A and Mix 13 - Group 9) commenced at a stage where samples had gained a significant proportion of their ultimate strength i.e. 90 days of age. Longer age curing of 91 days was required for the three mixes as they were slow setting and hardening mixtures. The optimum liquid / solid ratios for the mixtures in Group 2A was 19% and that of Groups 2 and 9 was 17%.

Two sets of six specimens were used in the experiment for each mix. On completion of curing for the first set 'A' (R_A), the specimens were placed in an environmental chamber and subjected to ten freeze-thaw cycles. Each freeze-thaw cycle lasted 24 hours with a starting temperature of 20°C, to -1°C in two and half hours, maintained cool at -1°C for four hours, reduced to -17½°C in three hours, maintained cool at -17½°C for six hours

and warm again to 20°C in eight and half hours. The relative humidity of the cabinet was set at 100%. After completion of the tenth freeze-thaw cycle, specimen 'A' was returned for 1 day curing to ensure complete thawing. The compressive strengths of set 'A' and the control set 'B' (R_B) were measured and the mean value of the strength for each set compared.

8.3 High Pressure Permeability Test on Preferred Mixes

This experimental work was similar to the one adopted by (Ganjian, Eshmaiel et al. 2004; Ganjian, Eshmaiel et al. 2006), where a modified Hoek cell was used to measure the permeability of the hydraulic paste in preventing the flow of fluid around the sides of the sample. Figure 8.1 shows the layout of the test. Methods of compaction of the samples was like that used for the compressive strength tests on the hydraulic cube pastes to achieve better density. The liquid / solid ratios were based on the ultimate strengths. A gang of three 50mm cylindrical moulds was used to produce cylindrical shaped specimens with 55mm diameter and at least 50mm in length.



Figure 8.1: High Pressure Flow Test Layout

After the specimen had been installed in the triaxial cell and bolts on the external framework tightened, an oil pressure of up to 40 bars was applied. Flow around the

specimen was prevented by maintaining the high pressure of hydraulic oil inside the flexible membrane. The pressure was maintained with the ramp pump by adjusting the weight on the balance unit. The water pressure valve was set at 30 bars, 10 atmospheres lower than the oil pressure. The permeability of the specimen was based on measuring the time it took to collect 25 ml of water passing through the Hoek cell.

Darcy's Law Equation was used to determine the intrinsic permeability of the hydraulic pastes in this research study.

$$v = \frac{Q}{A} = \frac{kdP}{\mu dL}$$

Where:

re: \mathcal{V} = velocity of flow (m/s)

Q = volume of flow rate (m³/s)

dP = pressure loss (Pa) over flow path of length dL (m)

 μ = velocity of fluid (Pa.s)

k = intrinsic permeability of the porous medium (m²/s)

A = cross sectional area of hydraulic paste specimen (m²)

The intrinsic permeability (k) is related to the hydraulic conductivity (K) and is in equation 6.

$$K = \frac{kg\rho}{\mu} \qquad \qquad [E]$$

[Equation 8.2]

[Equation 8.1]

Where:

K = coefficient of permeability (m/s)

 ρ = density of water (kg/m³)

g = gravitational acceleration (m²/s)
8.4 Resistance to Freezing and Thawing Results

After the specimens of the top three mixes [Mix 1 – Group 2 (PG20/RAF20/GGBS60), Mix 13 – Group 2A (V-B5G10/RAF30/GGBS60) and Mix 13 – Group 9 (PG10/QWD30/GGBS60)] were examined having gone through the tenth freeze-thaw cycle, they showed no damage of scaling and cracking. The lack of scaling and cracking may have been due to the presence of smaller particle size in the components. It was assumed that the smaller particle size present improved the packing. This improved packing may have provided better matrix, served as fillers and increased compaction of the components to eliminate air voids.

Review of research work by (Mangulkar and Jamkar 2013) showed particle packing models are based on the concept that voids between larger particles would be filled by smaller particles thereby reducing the volume of voids or increasing the packing density. In other words, one of the major effects of particle packing theory is its minimization of voids or maximization of the packing density of the components.

(Sarkkinen, Kujala and Gehör 2019) applied the same experimental standard (CEN/TS) used in this research study to compare alkali activated composite and ordinary portland cement as stabilization agents. Their results indicate that there was no damage in the specimens due to the freeze-thaw cycles because the compressive strength and density increased after the 10 cycles. The increase in strength and density was likely caused by an increase in binder products formed due to extra moisture and high temperatures during the warmer cycle period.

The results of the three mixes (Table 8.1) showed a loss in strength below the strength for the control specimens. Mix 1 of Group 2 and Mix 13 of Group 2A had retained ratios of approximately 93%. Mix 13 of Group 9 had retained ratio greater than 98% when compared to Mix 1 of Group 2 and Mix 13 of Group 2A. (Hamoush et al. 2011) concluded in their research work on freezing and thawing durability of very high strength concrete that a good freeze - thaw resistance should be a durability factor greater than 85% to avoid freeze/thaw damage.

	9	Set 'A'	9	Durability	
Mix	91d (MPa)	Density Kg/m3	91d (MPa)	Density Kg/m3	Factor R₄/R _B (%)
Mix 1 Group 2 (PG20/RAF20/GGBS60) Mix 13 Group 2A (V-B5G10/RAF30/GGBS60) Mix 13 Group 9 (PG10/QWD30/GGBS60)	36.43 36.13 36.75	1895 2002 1963	38.99 38.67 37.41	1930 2014 1980	93.43 93.44 98.23

Table 8.1: Resistance to freezing and thawing on Mixes 1 (Group 2), Mix 13 (Group 2A) and Mix 13 (Group 9)

8.5 High Pressure Flow Test Results

The surface skin of concrete is the first line of defence against the ingress of aggressive agents such as chlorides, sulphates and carbon dioxide. For this reason, there is an increasing awareness of its importance for durability of concrete (Claisse, Peter A., Ganjian and Adham 2003). Results of the flow test at 90 days showed that Mix 1 of Group 2 had lower coefficient of permeability of 2.40E-09 compared to Mix 13 of Group 2A (1.336E-08). The flow test results revealed that Mix 13 of Group 9 had a slightly lower coefficient of permeability of 5.283E-10 when compared to Mix 1 (Group 2) and Mix 13 (Group 2A). When the results of 28 and 90 days test were compared to all three mixes, the trends revealed similar outcomes. There was a decrease in the flow of liquid as the specimens age. The high permeability for the three mixes generally may have been due to the absence of aggregates in the constituents. (Ganjian, Eshmaiel et al. 2006) stated the properties of an ideal barrier system should have a low permeability of not less than 10⁻⁹ m/s in the UK. Figure 8.2 shows the results of the 28 and 90 days tests carried out on Group 2 Mix 1 (PG20/RAF20/GGBS60), Group 2A Mix 13 (V-B5G10/RAF30/GGBS60) and Group 9 Mix 13 (PG10/QWD30/GGBS60).



High Pressure Permeability of Preferred Mixes At 28 And 90 Days

Figure 8.2: High Pressure Permeability of Preferred Mixes

8.6 Choosing the Optimum Blend

The medium to long-term compressive strength results for all top three mixes performed exceptionally well. Figure 8.3 shows the results of the 7, 28 and 90 days tests. Mix 13 of Group 9 (30MPa) obtained the highest compressive strength at 28 days when compared to Mix 1 of Group 2 (22MPa) and Mix 13 of Group 2A (28MPa). At 90 days when the ultimate strength of the specimen was achieved, the results of Mix 13 on Group 9 was 38MPa. The strongest hydraulic binder at ultimate strength was Mix 1 of Group 2 (41MPa) followed by Mix 13 of Group 2A (40MPa).

In addition to the long-term compressive strength results obtained, the results of two durability tests mentioned in this chapter had great influence in the recommendation of the optimum blend for this research study. Results of the freeze-thaw experiments revealed that Mix 13 of Group 9 had a retained strength ratio greater than 98% when compared to Mix 1 of Group 2 (93%) and Mix 13 of Group 2A (94%).



Figure 8.3: Compressive strength at 9, 28 and 90 days of top three mixes

Permeability in concrete generally controls the rate of entry for lots of harmful substances which could have a detrimental effect on the environment. The findings of this study reasonably concluded that the more permeable the hydraulic binder was, the higher susceptibility to freeze-thaw cycles. The freeze-thaw and high-pressure permeability test results of Mix 13 of Group 9 had highest retained strength ratio and lowest permeability respectively when compared to the other two mixes.

Based on the analysis set out in this section, it was recommended that Mix 13 of Group 9 with the proportions of 10%PG, 30%QWD and 60%GGBS as the optimum blend (also referred to as the '**Warwickshire Blend'**) for this research study. Not only did the 'Warwickshire Blend' out-perform the other two mixes on long-term durability, it achieved the strongest compressive strengths at 7 and 28 days approximately (6MPa and 30MPa respectively) when compared to Mix 1 of Group 2 (2MPa and 21MPa) and Mix 13 of Group 2A (2MPa and 28MPa). Strength development of the 'Warwickshire Blend' at 7 days was over 14% when compared to the Mix 1 and 13 of Groups 2 and 2A at 5%. At 28 days, the strength development was 75% compared to 50 and 69% for Groups 2 and 2A respectively. There was a slight decrease of approximately 5% in strength development from 90 to 180 days on Mix 1 of Group 2. This slight decrease may have been the effect of variability of the by-product materials obtained at various stages in the research. Highlighted in bold in Table 8.2 are the compressive strength results up to 180 days for the top three mixes. Figure 8.4 shows the percentage strength development of the top three mixes.

Table 8.2: Strength development of selected mixes on Groups 2, 2A and 9

Group Mix Design Reference			L/S Datio				
Group	wix Design Reference	7	14	28	90	180	
	Mix 1 - PG20/RAF20/GGBS60	2	13	21	41	39	0.17
2	Mix 4 - PG5/RAF47.50/GGBS47.50	2	9	19	30	38	0.17
2	Mix 6 - PG15/RAF37.50/GGBS47.50	2	9	18	35	36	0.17
	Mix 13 - PG10/RAF30/GGBS60	2	18	25	37	39	0.17
	Mix 1 - V-B5G20/RAF20/GGBS60	3	12	26	33	35	0.19
24	Mix 4 - V-B5G5/RAF47.50/GGBS47.50	1	19	21	31	40	0.19
ZA	Mix 6 - V-B5G15/RAF37.50/GGBS47.50	2	12	24	33	35	0.19
	Mix 13 - V-B5G10/RAF30/GGBS60	2	13	28	40	41	0.19
	Mix 1 - PG20/QWD20/GGBS60	2	10	26	34	37	0.17
0	Mix 4 - PG5/QWD47.50/GGBS47.50	2	8	22	32	40	0.17
9	Mix 6 - PG15/QWD37.50/GGBS47.50	2	6	24	30	33	0.17
	Mix 13 - PG10/QWD30/GGBS60	6	13	30	38	41	0.17



Figure 8.4: Percentage of strength development of Mix 1 (Group 2), Mix 13 (Group 2A) and Mix 13 (Group 9)

CHAPTER 9 – PHASE FOUR EXPERIMENTAL TESTS ON SUSTAINABLE CONCRETE

9.1 Introduction

This chapter describes tests that were carried out on sustainable concrete made from the 'Warwickshire Blend' (WB). Analysis of the results were based on compressive strength, absorption tests, workability and the hydration process involved with the specimens.

9.2 Sustainable Concrete

Laboratory tests to determine the mechanical properties of the sustainable concrete was conducted using three different approaches, namely:

- i. concrete mixtures comprising of the WB (PG10/QWD30/GGBS60) with natural fines and coarse aggregates;
- ii. concrete mixtures comprising of WB with cement as a supplementary material, combined with natural fines and coarse aggregates; and
- iii. concrete mixtures comprising of the WB with crushed recycled concrete as fines / coarse aggregates and WB with combination of crushed recycled concrete and natural fines / coarse aggregates.
- 9.2.1 WB Concrete with Natural Fines and Coarse Aggregates

(Jackson and Dhir 1996), stated, for a given mix proportion, the influence of cement on concrete strength is determined by its fineness and chemical composition through the process of hydration. The gain in strength is most associated with the fineness of the cement.

(Neville 2011) described cement in general sense as a material with adhesive and cohesive properties which makes it capable of bonding mineral fragments into a compact whole.

As the research conducted in this study was to develop a hydraulically bound cementitious

material for applications in road(base), foundation and sub-grade in pavement engineering, it is important to define what hydraulically bound mixture is in this context. In accordance with (EN, BS 13286 - 40 2003), hydraulically bound material is a mixture that hardens by hydraulic and / or pozzolanic, sulphatic and / or carbonic reaction, which usually has a workability to suit compaction by rolling and which is generally used in bases, sub-bases and capping layers.

9.2.1.1 Mix Proportions for Experiments on WB Concrete with Natural Fine and Coarse Aggregate

The ternary combination of the WB has cohesive properties that enabled it to perform as a binder. Table 9.1 gives detail of the mix proportions together with the contents of binder dosages for the different liquid / solid ratios on the WB with natural fines and coarse aggregates. The specimens were dry cured, wrapped in a plastic cling film and placed in a temperature control room at (20+/-2) °C. The specimens were cured for 7, 28 and 90 days.

Table 9.1: Mix proportions	of concrete mixtures -	- WB with r	natural fines and
coarse aggregates			

	Mix Proportions (Kg/m3)								Slump
Mix Design Ref.	PG	QWD	QWD GGBS OPC Water San		Sand	Coarse 10 mm	L/S	(mm)	
OPC100 (L/S 0.42)	-	-	-	423.3	178	741	1058	0.42	0
OPC100 (L/S 0.50)	-	-	-	417.4	209	730	1043	0.50	55
PG10/QWD30/GGBS60 (L/S 0.42)	42.3	127.0	254.0	-	178	741	1058	0.42	0
PG10/QWD30/GGBS60 (L/S 0.50)	41.7	125.2	250.4	-	209	730	1043	0.50	60

9.2.2 WB Concrete with Cement as Supplementary Material

9.2.2.1 Reviews on Supplementary Mixes

A study to investigate the optimum level of replacement of GGBS on compressive strength of concrete was carried out by (Oner and Akyuz 2007). GGBS was added in an amount equivalent to 0%, 15%, 30%, 50%, 70%, 90% and 110% of cement contents of control concretes with 250, 300, 350 and 400kg/m³ dosages. The results revealed that the early age strength values of GGBS concrete mixtures were lower than the control mixtures. As the specimens age, the strength values of the GGBS concrete mixtures increase more

than the control mixtures. It was concluded that the optimum level of GGBS content for maximising strength is between 55 – 59% of the total binder content.

(Neville 2011), stated that a cement consisting of Portland cement and one or more appropriate inorganic materials will be called blended cement. (Neville 2011), went on further to say that all the materials with which we are concerned have in common that they contribute to the strength-gaining properties of the cement. Some of these materials are cementitious in themselves, some have latent cementitious properties like the QWD and GGBS in the WB, and this contributes heavily to the strength of the concrete primarily through their chemical and physical behaviours.

(Sharma and Khan 2016), reviewed papers on the effect of different supplementary cementitious materials (Fly Ash – FA, Silica Fume – SF, Metakaolin – MK and GGBS) on mechanical and durability of concrete. It was revealed in the literature that GGBS was found to increase the mechanical and durability properties at later age depending upon the level of replacement. It was noted that 80% replacement of cement by GGBS reduces strength at all ages. For replacement between 40% and 60%, strength was greater than the control mix after 28 days of curing. They observed that, increase in slag replacement by weight at certain levels, decreases the strength of concrete in short-term as compared to control concrete, but long-term strength of concrete containing slag exhibits same or more than that of the control mix. Their finding compliments those of (Oner and Akyuz 2007).

(Johari et al. 2011), investigated in their study the influence of supplementary cementitious materials (SCM), namely SF, MK, FA and GGBS on properties of high strength concrete (HSC). Tests to determine workability, compressive strength, porosity and elastic modulus were assessed. It was found that the presence of GGBS influenced the long-term performance of the concrete. The early strength development of the GGBS concrete was lower than that of PC concrete, particularly at higher levels of replacement. This they concluded, may have been due to the dilution effect as part of the cement was replaced as well as the slower reactivity of GGBS. Concrete with a 60% GGBS replacement showed very low strength at 1 day, and this may be attributed to the excess retardation in setting and hardening of the concrete. Strengths of the mixes containing 20%, 40% and 60% GGBS at 1 day age were 72%, 45% and 4.6% respectively. At higher replacement levels

of 40% and 60% and longer curing periods, the GGBS concrete obtained strengths greater than or comparable to the control specimens. The maximum relative strength achieved were 104% and 102% respectively at 90 days.

9.2.2.2 Mix Proportions for Experiments on Supplementary Mixes

A laboratory study to identify the mixture with the minimum level of replacement with PC and obtain the highest compressive strength of the WB mixture was carried out. The percentage by weight of the three different components in the WB mixture were proportionately altered to allow the partial replacement in an amount of 5%, 10%, 20% and 30% of PC. In other words, the WB mixture was partially replaced with cement. The study of the mixes were carried out using liquid / solid ratios of 0.42 and 0.50. Control mixes of 100% PC was used to compare the results. The specimens were cured in a bath of water and were later tested for compressive strengths for 7 and 28 days. Table 9.2 gives detail of the mix proportions together with the contents of cement / binder dosages for the different liquid / solid ratios.

	Mix Proportions (Kg/m3)								Clump
Mix Design Ref.	PG	QWD	GGBS	OPC	Water	Sand	Coarse 10 mm	L/S	(mm)
OPC100 (L/S 0.42)	-	-	-	423.3	178	741	1058	0.42	0
OPC100 (L/S 0.50)	-	-	-	417.4	209	730	1043	0.50	55
PG9.5/QWD28.5/GGBS57/OPC5 (L/S 0.42)	40.2	120.6	241.3	21.2	178	741	1058	0.42	0
PG9.5/QWD28.5/GGBS57/OPC5 (L/S 0.50)	39.7	119.0	237.9	20.8	209	730	1043	0.50	80
PG9/QWD27/GGBS54/OPC10 (L/S 0.42)	38.1	114.3	228.6	42.3	178	741	1058	0.42	0
PG9/QWD27/GGBS54/OPC10 (L/S 0.50)	37.6	112.7	225.4	41.7	209	730	1043	0.50	100
PG8/QWD24/GGBS48/OPC20 (L/S 0.42)	33.9	101.6	203.2	84.6	178	741	1058	0.42	15
PG8/QWD24/GGBS48/OPC20 (L/S 0.50)	33.4	100.2	200.3	83.5	209	730	1043	0.50	110
PG7/QWD21/GGBS42/OPC30 (L/S 0.42)	29.6	88.9	177.8	127.0	178	741	1058	0.42	25
PG7/QWD21/GGBS42/OPC30 (L/S 0.50)	29.2	87.7	175.3	125.2	209	730	1043	0.50	120

Table 9.2: Mix proportions of concrete mixtures – WB with supplementary mixes

9.2.3 WB Concrete with Crushed Recycled Concrete

9.2.3.1 Reviews on Recycled Concrete Aggregates

Extensive research has been carried out on the mechanical performance and long-term durability of recycled concrete as fine and coarse aggregates. (Xiao et al. 2012), reported

that China's consumption of cement is 820 million tons and accounts for 55% of the world. Experiment results on the compressive strength of recycled aggregate concrete (RAC) mentioned in (Xiao et al. 2012) study indicated that the amount of recycled coarse aggregate (RCA) has remarkable influence on the compressive strength of concrete. In general, the strength decreases with increase of the RCA content. However, if the RCA content is less than 30%, the influence on the strength is less obvious.

An experimental study carried out by (Rahman, Hamdam and Zaidi 2009), whereby 100% of recycled aggregate (RA) used in concrete mix to replace the natural coarse aggregate in concrete with a target strength of 25MPa at 28 days. The results were compared to concrete with natural aggregates as control mix. The compressive strengths for all the six different mixes used in the experiments achieved the target strength of 25MPa for the RAC.

A study carried out by (Ann et al. 2008), investigated recycled aggregates from crushed concrete to replace natural aggregate. PFA and GGBS were partially replaced for cement in binder to enhance the concrete properties in terms of long-term durability. The results revealed that the compressive strength of concrete containing recycled aggregate at 7, 28, 90 and 180 days was lower than the control mixtures, and recovered by replacing 30%PFA and 65%GGBS. The strength achieved for the concrete containing recycled aggregate at 180 days was 26MPa when compared to the three other mixes (recycled – 30%PFA and recycled – 65%GGBS) which were over 30MPa.

Laboratory experiments were conducted by (Mas et al. 2012), on the influence of replacing natural aggregate with mixed recycled aggregate (MRA) in the mix design of a non-structural concrete. It was concluded in the study that a decrease in strength takes place when the proportion of MRA increases. The loss of strength is correspondingly higher as the strength increases. The loss of strength compared with the control mix is less at 90 days than at 7 and 28 days. Additions of MRA of between 20 - 25% decreases in strength less than 15%.

9.2.3.2 Mix Proportions for Experiments on WB Concrete with Recycled Concrete as Fines / Coarse Aggregates and Combination of Recycled Concrete and Natural Aggregates The laboratory experiments conducted involved the use of the WB with crushed recycled concrete to replace natural fine and coarse aggregate in one mix design (RAC-WB) and, another mix design (BAC-WB) the combination of recycled concrete and natural fine and coarse aggregate. Two control mixes were used to compare the results. The first control mix with 100% cement with crushed recycled concrete as fine and coarse aggregate (RAC-100%OPC). The second control mix with 100% cement, blending of recycled concrete and natural aggregates (BAC-100%OPC). The design of the RAC mix was based on (ACI 211 Standard, 1996), and that of BAC was based on Equivalent Mortar Volume (EMV) method. The specimens were cured in a bath of water and were later tested for compressive strengths for 7, 28 and 90 days. Table 9.3 gives detail of the mix proportions used in this experiment.

Mix Properties	RAC	BAC
Cement (kg/m³)	507	364
Recycled Fine Aggregates (kg/m ³)		
0.075 - 0.57 mm	267	153
0.57 - 2.47 mm	178	102
2.47 - 4.75 mm	89	51
Natural Fine Aggregates (kg/m ³)		
0.075 - 0.57 mm		102
0.57 - 2.47 mm		68
2.47 - 4.75 mm		34
Recycled Coarse Aggregates (kg/m ³)		
4.75 - 10 mm	528	528
10 - 14 mm	226	226
Natural Coarse Aggregates (kg/m ³)		
4.75 - 10 mm		345
10 - 14 mm		148
Water (kg/m³)	213	153
Superplasticizer (kg/m³)	1.52	7.28

Table 9.3: Mix proportions of materials

9.2.3.3 Source of Recycled Aggregate Concrete and Preparation of Samples

The recycled concrete was sourced locally from Litecast Limited, located at Camphill in

Nuneaton, North Warwickshire. Litecast is a leading supplier for precast concrete flooring solutions and the concrete wastes are produced as a result of defective concrete floor beams during the manufacturing process.

Preparations of the samples to achieve different gradings of the fine and coarse aggregate was carried out by using the Avery Denison 2000 machine. The large samples of partially crushed concrete were placed in a 15 - 20mm thick metal ring, supported on a 50mm thick base plate and the hydraulic pressure was set at 450KN. A constant rate of loading at 5KN/s was applied to the samples. The 450KN was set to ensure depression of approximately 70 – 90mm of the samples, which allowed the concrete to be crushed into manageable pieces used for the different gradings. Figures 9.1 and 9.2 show the set-up before and after the crushing of the samples.



Figure 9.1: Preparation of equipment before crushing



Figure 9.2: Crushed samples

9.3 Experimental Results of Sustainable Concrete

9.3.1 Results of WB Concrete with Natural Fines and Coarse Aggregates

In general, all the mixes had low compressive strengths compared to the OPC concrete. Results of the compressive strength show that the mixture with liquid / solid ratio of 0.42 had the highest strengths at 7, 28 and 90 days compared to the mixture with 0.50 l/s ratio. The strongest strength recorded for the WB concrete with natural fines and coarse aggregates was 26MPa at 90 days, compared to 46MPa for the control mix at the same I/s ratio of 0.42. This showed that the amount of water in the mixes of 0.42 I/s ratio was enough for complete hydration of the concrete to take place. This finding is in agreement with a similar study carried out by (Shyam Prakash and Rao 2016), where they concluded that cement with a liquid / solid ration of 0.45 has higher compressive strength compared with liquid / solid ratio of 0.50. The higher strength was attributed to the water absorption of the quarry dust. Table 9.4 and Figures 9.3 show the results of the 7, 28 and 90 days tests.

Mix Dosign Rof		Strength at Days (MPa)					
wix Design Kei.	7	28	90	(Kg/m³)			
OPC100 (L/S 0.42)	41	50	53	2332			
PG10/QWD30/GGBS60 (L/S 0.42)	2	13	26	2331			
OPC100 (L/S 0.50)	25	42	46	2311			

8

20

2322

1

Table 9.4: Compressive Strength of WB and Control Mix at 7, 28 and 90 days

PG10/QWD30/GGBS60 (L/S 0.50)



Figure 9.3: Comparison of WB concrete with control mix

9.3.2 Results on WB Concrete with Cement as Supplementary Material

All the mixes had low compressive strengths compared to the OPC concrete. Results of the compressive strength show that mixtures with liquid / solid ratios of 0.42 had the highest strengths at 7, 28 and 90 days compared to the mixtures with 0.50 l/s ratio. Table 9.5, Figures 9.4 and 9.5 show the results of the 7, 28 and 90 days tests.

Table 9.5: Compressive Strength of Supplementary Mixes at 7, 28 and 90 days

	Str	Density		
Mix Design Ket.	7	28	90	(Kg/m³)
OPC100 (L/S 0.42)	41	49	55	2377
OPC100 (L/S 0.50)	30	37	43	2303
PG9.5/QWD28.5/GGBS57/OPC5 (L/S 0.42)	7	11	13	2358
PG9.5/QWD28.5/GGBS57/OPC5 (L/S 0.50)	6	7	9	2325
PG9/QWD27/GGBS54/OPC10 (L/S 0.42)	9	12	13	2347
PG9/QWD27/GGBS54/OPC10 (L/S 0.50)	6	8	9	2316
PG8/QWD24/GGBS48/OPC20 (L/S 0.42)	11	19	22	2337
PG8/QWD24/GGBS48/OPC20 (L/S 0.50)	10	17	21	2325
PG7/QWD21/GGBS42/OPC30 (L/S 0.42)	13	23	25	2346
PG7/QWD21/GGBS42/OPC30 (L/S 0.50)	10	19	23	2333



Figure 9.4: Comparison of Supplementary mixes at 0.42 ratio



Figure 9.5: Comparison of Supplementary mixes at 0.50 ratio

The trend in strength development for mixtures with 0.42 l/s ratios was linear for both 7 and 28 days. Mixtures with 0.50 l/s ratios on the other hand showed a linear trend in strength development at 28 days only. In general, there was an increase in strength development as the specimen ages. It can be said from the results that, as the level of replacement with OPC increases, there was an increase in compressive strength on the WB concrete with cement as supplementary material.

It was observed from the experimental results that a minimum level of replacement of 10% OPC can obtain compressive strength of approximately 10MPa (PG9/QWD27/GGBS54/OPC10) at 7 days with liquid / solid ratio of 0.42. Replacement of 20% OPC can obtain strength of approximately 20MPa (PG8/QWD24/GGBS48/OPC20) at 28 days with liquid / solid ratio of 0.42.

9.3.3 Results on WB Concrete with Recycled Concrete as Fines / Coarse Aggregates and Combination of Recycled Concrete and Natural Aggregates

9.3.3.1 Compressive Strength

The experimental test results revealed that the control mix of BAC-100%OPC had highest compressive strengths at 7 and 28 days, followed by RAC-100%OPC (no tests were carried out at 90 days for the control mixes). The compressive strengths of the WB with recycled concrete as fines and coarse aggregates (RAC-WB) were 15, 19 and 23MPa at 7, 28 and 90 days respectively. The compressive strengths of the WB with the combination of recycled aggregate concrete and natural aggregate (BAC-WB) were 18, 24 and 27MPa at 7, 28 and 90 days respectively. However, direct comparisons cannot be made to the control mixes in strength development by age on the WB mixtures in the experimental tests, as strength development of RAC-WB and BAC-WB concrete was delayed due to their pozzolanic properties. Even though a direct comparison cannot be made, it is reasonable to conclude the results of the tests on their relative strengths at 28 days. The relative strength of the control mixtures was achieved at 28 days, and that of the WB mixtures 90 days of age. Table 9.6 and Figures 9.6 show the results of the 7, 28 and 90 days tests.

In comparing the trends of the control mixes to that of the WB mixes, mixtures with the combination of recycled concrete aggregate and natural aggregate performed better than mixtures with 100% crushed concrete as fines and coarse aggregates. These findings agree with that concluded in a separate study carried out by (Xiao et al. 2012). (Xiao et al. 2012), indicated in their study that in general, the strength decreases with increase of the crushed recycled concrete content. The mix (RAC-WB) with 100% crushed recycled concrete as fines and coarse aggregate had lower strengths at 7, 28 and 90 days when compared to the mix (BAC-WB) that had combination of recycled concrete and natural fines / coarse aggregates.

Mix Decign Ref	Stre	Density		
Witz Design Ref.	7	28	90	(Kg/m3)
RAC-100%OPC	46	56	-	-
BAC-100%OPC	55	63	-	-
RAC-WB	15	19	23	2174
BAC-WB	18	24	27	2338

Table 9.6: Compressive Strength of RAC / BAC and Control Mix at 7, 28 and 90	0 da	ay
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Figure 9.6: Comparison of RAC and BAC mixes

9.3.3.2 Water Absorption

The laboratory experiments conducted for water absorption (WA) involved the use of WB with recycled concrete as fines and coarse aggregates (RAC-WB) and WB with the combination of recycled concrete and natural fine and coarse aggregates (BAC-WB). The laboratory tests were carried out in accordance with BS 1881 – 122:2011. There were six cylindrical specimens for each mix design, with the dimensions 200mm high by 100mm diameter. The specimens were cured in a bath of water for 28 days (X3 gang) and 90 days (X3 gang). After the respective curing periods, the specimens were placed in an oven and left to dry for 72 hours with the oven temperature set at 105° C (+/- 5° C). On removal of the specimens on the third day, they were put in an airtight vessel and left to cool for 24 hours.

For the water absorption measurement, the weight of each specimen was recorded before they were completely immersed in water, at a depth of 25mm (+/- 5mm) above the top of the specimens. The specimens were then removed after 30 minutes to measure the water absorption. After removal of the specimens from the water, excess water was removed from the surface with a paper towel to dry. The weight of each specimen was recorded.

The water absorption for the RAC-WB mix was recorded approximately as 5.5% for 28 days and 90 days age of curing. The water absorption for the BAC-WB mix design on the other hand were approximately 3.2% and 3% for 28 days and 90 days age of curing respectively. The absorption rate of the specimens was obtained using cumulative immersion periods at 10, 30, 60 and 120 minutes and the results for the 28 days and 90 days are shown in Figure 9.7 and Figure 9.8. In accordance with Volume 2 – Series 800 Notes for Guidance (NG) on the Specification for Highway Works, the supplier is required to monitor the WA values as part of the system of factory production control. The value for the aggregate used should be stated and this value may be used to provide a baseline for routine WA tests. Volume 2 – Series 1000 NG on the Specification for Highway Works mentioned that if the result from these tests exceeds the declared value (d) by more than 0.5 i.e., > (d + 0.5)%, further investigation will be required. Sandstone and limestone aggregates are usually used in road construction and the WA for these aggregates are in the range of between 3 - 20% and 3 - 15% respectively. The experimental results for the two mix designs (RAC-WB and BAC-WB) fall within the range of the aggregates assumed to be mentioned in this section of the report, therefore in accordance with the criteria set out in the guidance notes referred to in the specifications.



Figure 9.7: Absorption rate of WB with recycled concrete as fines and coarse aggregates



Figure 9.8: Absorption rate of WB with combination of recycled concrete and natural fines and coarse aggregates

A study conducted by (Pavía and Condren 2008), reported on water absorption rate at 28 days cycles as GGBS was used to replace PC by 30% and 50% by weight over a four months period. The investigation revealed that for all the samples, including the control specimen (PC100% by weight) that as time increases, the water absorbed increases. It was noticeable that increase in the GGBS replacement, reduces the water absorbed. The average increase of the water absorbed for the three specimens: PC100%, GGBS30% and GGBS50% were 18.65, 12.65 and 8.26 respectively. The report concluded that GGBS mixes showed the smallest progressive rise in water absorption and this may be due to the dense and strong microstructure of the interfacial aggregate / binder transition zone.

The findings of the experimental tests were the same as those encountered by Pavia et al. 2008, whereby the water absorption rate increases with time. There was a slightly higher increase in the absorption rate for the 90 days specimens to that of the 28 days on the RAC-WB mix. For the BAC-WB mix, it was observed that the absorption was slightly lower for the 90 days specimens compared to the 28 days.

9.4 Workability

A slump test in accordance with BS 1881: Part 102 was investigated in this research study for measuring the workability of the sustainable concrete mixtures. The slump test results obtained from the experiments are shown in Tables 9.1 and 9.2.

(Jackson and Dhir 1996), defined workability as having three main characteristics: i) consistency; ii) mobility; and iii) compactability. They explained consistency as a measure of wetness of fluidity, mobility the ease with which a mix can flow into a framework or mould and compactability the ease a mix can be fully compacted to remove all trapped air. (Jackson and Dhir 1996), went on further to say that the test on workability, does not measure the individual characteristics, it only provides a guidance on the workability of a mix. They mentioned many factors that may affect workability, some can be summarised as: i) the constituent of materials used in the mix; ii) method of compaction; iii) physical properties of the materials with regard to shape, roughness, specific surface; and iv) water content.

It can be seen from the slump tests carried out on the mixes in Tables 9.1 and 9.2 that all the mixtures with liquid / solid ratios of 0.42 had zero slump, except for mixtures where the cement content by weight were 20 and 30 percentage. None of the mixtures with 0.50 liquid / solid ratio observed a zero slump. It can be said from the observations made from the experimental results carried out in this study that, as the percentage of cement increases, the slump increases. This may have been as the results of the physical properties of the cement with regards to shape and specific surface.

9.5 Analysis of Sustainable Concrete

It can be concluded from the laboratory investigation carried out in this research study that the WB concrete, on its own can perform in terms of mechanical stability without the aid of any form of supplementary materials, achieving a compressive strength of 26MPa at 90 days.

Oxides	PC (%)	PG (%)	QWD (%)	GGBS (%)
SiO ₂	17 - 25	0.96	34.00	33.23
TiO ₂	-	0.03	1.80	1.09
AI_2O_3	3 - 8	0.31	12.00	13.14
Fe ₂ O ₃	0.5 - 6	0.27	12.00	0.52
MnO	-	0.00	0.41	0.33
MgO	0.5 - 4	0.13	5.30	8.92
CaO	60 - 67	40.00	14.00	39.76
Na ₂ O	-	0.02	2.60	0.21
K ₂ O	-	0.09	0.87	0.45
P_2O_5	-	0.02	0.56	-
SO₃	2 - 3.5	52.00	0.99	1.12

Table 9.7: Chemical oxides of materials (PC obtained from (Neville 2011))

Some of the key factors that may have influenced the strength were mentioned in Section 7.8. One of the main factors contributed to the success of the WB concrete, either by its own; the use of supplementary materials such as cement to enhance its performance; or the use of recycled concrete as fines and coarse aggregates; is the correct water content used to achieve full compaction and maximum density. Chemical compositions of the materials followed by physical properties may have been the other factors. The chemical compositions and physical properties of the binders used in this research study are shown in Table 9.7 and Figure 9.9. The chemical compositions shown in Table 9.7 for cement were obtained from (Neville 2011).

There are similarities between the materials with regard to the proportions of SiO₂ present in the PC, QWD and GGBS. For the PC, PG and GGBS, the similarities between the materials are the presence of CaO, also the dominant oxide in the binder constituents. As for the pozzolanic materials: QWD and GGBS, there similarities are with regard to the proportions of SiO₂ and Al₂O₃. The QWD in the matrix contains a very small amount of CaO. It was assumed that the PC and PG on the other hand contributed a considerable amount of CaO to the reaction processes. (Jackson and Dhir 1996), stated that the combination of such materials produced hydrated cementitious products by undergoing a pozzolanic reaction with calcium hydroxide liberated by the PC.

It is assumed that the presence of the dominant oxides of CaO, coupled with the pozzolanic activities helped the mechanical and sorptivity performance of the WB concrete, not only as a stand-alone material, but also incorporated with recycled concrete and cement as partial replacement to the WB.



Figure 9.9: Particle size distribution of PG, QWD and GGBS

CHAPTER 10 - CONCLUSIONS

10.1 Research Questions

To conclude this research project, the following questions below were asked and answers provided. This will be followed from conclusions that can be drawn from the results obtained from the study.

- i. Why this area of study, into the use of industrial by-products and its potential use in Highways Engineering?
- ii. What was the aim of the research project?
- iii. How was the aim achieved and what was the strategy used to achieve a positive research outcome?
- iv. What were the objectives, and were they met and how?
- v. What were the parameters of the research? Where they realistic?
- vi. What is the author's contribution to knowledge?
- vii. What evidence has the author found that gives him a sense of the underlaying philosophy?
- viii. How were the three pillars of sustainable development met?

The answers to the above questions have been summarised in the order in which they have been presented:

- As a practicing Chartered Civil Engineer and having worked in the construction industry for over twenty years, the lack of interest by stakeholders, and the potential positive environmental impact the extensive use of industrial by-products could have, instigated the author to research in this area;
- ii. The research aim was to investigate feasibility of replacing cement fully with industrial by-products obtained within the United Kingdom.
- iii. There is no doubt that the outcome of the research will make a positive change to peoples' lives, as sustainable development is not just about meeting our own

needs, but meeting those of future generation's need. The positive outcome was due to the 'Methodology Overview' and 'Testing Methodology for By-product Suitability' adopted in the research study as detailed in Section 2.1 and 4.2 respectively.

- iv. All the objectives have been met and are detailed in Section 10.2.
- v. The scope of the research was reduced as the materials identified at the initial stages in Section 2.3 were too many. Limitations of the research were further reviewed by refining the top three mixes (Group 2 Mix 1 PG20/RAF20/GGBS60), (Group 2A Mix 13 V-B5G10/RAF30/GGBS60) and (Group 9 Mix 13 PG10/QWD30/GGBS60). As a result of the refinement, Mix 13 of Group 9 emerged as the novel mix or candidate materials referred to as WB (Section 8.6).
- vi. This research can fill the knowledge gap in the use of two waste gypsums (PG and V-B5G) and two quarry by-products (RAF and QWD) in combination with GGBS as full cement replacement in road (base) and foundation. Other contributions to knowledge include:
 - the design of experiment and analysis of the results were carried out using a statistical software package – Minitab 18. Extreme Vertices Design was used to set the boundaries of the components in each group for the design. The software has predictive capability that can be compared to the actual results obtained with high degree of accuracy;
 - the XRF results of separate batch of by-product materials were received over a three-year period. There were slight variations in the chemical and physical properties of the by-product materials obtained over this period; and
 - the use of EMV method for the sustainable concrete.
- vii. It is believed that the findings of this research achieved the aim and all the objectives set out. It was self-evident from the data obtained that cement can be <u>100%</u> replaced with industrial by-products, using the correct mix design, systematic manufacturing method and adequate curing adopted. Minitab 18 was

used for the design of experiment (Section 5.2), and the manufacturing and curing methods used outlined in Sections 5.4 to 5.6.

viii More than 70 definitions of sustainable development have been made and used or interpreted by different groups to suit their own goals. (Langston and Ding 2001), definitions of sustainable development abound since what constitutes development for one person may not be development or progress for another. The most widely used and popular definition is given in Section 1.2.3 (page 4), from the 'Brundtland Report.'

Even though the general benefits to the use of recycled and / or secondary waste minerals have been outlined in Section 1.4.4 as economic, environmental and social, the outcome of sustainability in this research study was analysed in the same way as detailed in Section 1.4.4.

Economic Impact

Based on the industrial by-products that constitute the optimum mix 'Warwickshire Blend' (PG10/QWD30/GGBS60), the use of this mix as a full cement replacement demonstrated in Section 8.6 as hydraulically bound mixture and Sections 9.2 and 9.3 as sustainable concrete will no doubt result in significant savings. This attribute deals with the performance of monetary or financial value. The PG and QWD are freely and locally available. The GGBS, when compared to PC is more expensive in price. This is due to its demand in the UK and costs associated to import from abroad. The use of this candidate mix does not only save costs associated in disposing of the PG and QWD to landfill sites, it completely removes the need to buy cement.

Environmental Impact

Environmental damage as a result of the manufacturing of cement have been outlined in Section 1.2. There is an impact on the environment from the extraction of natural resources, which usually leads to an increase in energy consumption, and this is a growing concern shared by many. The stress to the environment relates to the energy used to transport the raw materials and processes involved in manufacturing to the desired components. There is less impact on the environment in the use of the candidate materials, as they would have been diverted from landfill sites, increasing carbon dioxide savings through shorter distances from locally available source.

Social Impact

This attribute is analysed in terms of the enhancement to the quality of life. The social benefits derived from the use of not only the WB, but also the sustainable concrete of which the recycled fines and coarse aggregates were sourced from a local concrete manufacturing company. The PG, QWD and RCA had not been transported a significant distance from their source of production.

The use of the candidate mix identified in this study as alternative to cement and / or conventional concrete is not only cost effective, but also satisfies the following criteria:

- reducing haulage activities thereby reducing congestion;
- increasing road safety in reducing traffic on the road network; and
- reducing of noise and air pollution through shorter distance travelled.

10.2 Research Objectives

The specific objectives of the research were:

- to identify industrial by-product that are being taken to landfill sites and investigate their use in highways engineering;
- 2) to conduct laboratory tests to examine by-product suitability for use;
- to conduct strength tests on the eleven groups of mix design identified and durability tests on the top three mixes to obtain the novel blend; and
- 4) to add natural aggregate and recycled concrete aggregate in the novel blend to manufacture sustainable concrete.

10.2.1 Objective 1

Initial research in identifying waste minerals disposed of to landfill sites were conducted in two phases. Sectors from the Casting Federation, Cement, Chemical, Pharmaceutical, Steel, Mineral Aggregates and Recycled industries were initially approached via telephone interviews. The interviews via telephone were unsuccessful, and a second approach was adopted through email correspondence. Interests were expressed by some of the companies. They shared their environmental concerns in disposing of their wastes to landfills and offered to provide the materials used in the research for a good cause.

10.2.2 Objective 2

XRD and XRF experiments were used to determine industrial by-products' suitability. Further tests to determine alkalinity levels and pozzolanic indices for some of the materials were carried out. Materials identified to have good chemical and physical properties with distinct pozzolanic and / or cementitious characteristics were further explored in the research study.

10.2.3 Objective 3

In total, there were eleven different ternary groups that were analysed: PG/MHT/GGBS, PG/RAF/GGBS, V-B5G/RAF/GGBS, PG/GGBS/BPD-C, PG/GGBS/BPD-H, V-B5G/MHT/SSD, V-B5G/RAF/SSD, V-B5G/SSD/BPD-C, V-B5G/SSD/BPD-H, PG/QWD/GGBS and V-B5G/QWD/GGBS. The use of the statistical software package allowed for the design and analysis of the different constituents. Out of approximately three hundred mixes analysed on the hydraulic pastes, approximately 10% had compressive strengths greater than 20MPa at 28 days of age.

Conclusions from the ternary binder pastes analysed indicated that sulphate activated pozzolan mixtures exhibited greater strengths than alkali activated slag mixtures. The following observations were made in reviewing the mixes in the groups:

- due to the slower reactivity of the RAF, QWD and GGBS in the mixtures (which the author refers to as having 'Latent Pozzolanic Effect'), strength gain was extremely slow in mixtures containing two pozzolanic materials in the ternary components: PG/RAF/GGBS, V-B5G/RAF/GGBS, PG/QWD/GGBS and V-B5G/QWD/GGBS. After 28 days, and up to 90 days when the ultimate strength (i.e. 90 days age) off the hydraulic paste were achieved, strength increases with the presence of GGBS of up to the maximum percentage by weight (60%) used in the study. The chemical compositions (Section 4.4) and physical properties (Section 4.6) present in the different components may have influenced the mechanical stability results obtained in the study; and
- it can be possible to design full cement replacement materials for desired strengths based on the correct constituent of materials, adequate water content for the hydration process and correct curing method used. Variations in the water content in some of the mixes influenced the long-term compressive strength and can also achieve different compressive strengths in some of the mixtures at specified age. Table 6.27 of Section 6.5 shows the findings.

When reviewing the durability tests for the top three mixes (PG20/RAF20/GGBS60, V-B5G10/RAF30/GGBS60 and PG10/QWD30/GGBS60) identified to have had potential, the following results were concluded:

- results for the resistance to freezing and thawing showed a slight loss in strengths below the strength of the control specimens for all three mixes, with mix with the proportions PG10/QWD30/GGBS slightly higher compared to the other two; and
- the high pressure flow tests results showed that Mix 13 with the proportions 10%PG, 30%QWD and 60%GGBS had a lower coefficient of permeability of 5.28E-10 when compared to Mix 1 with the proportions 20%PG, 20%RAF and 60%GGBS, and Mix 13 with proportions 10%V-B5G, 30%RAF and 60%GGBS.

10.2.4 Objective 4

Three different approaches were used to obtain sustainable concrete based on the WB. WB concrete made of natural fines and coarse aggregates, WB concrete made of partial PC replacement, and WB concrete made of recycled concrete as fines and coarse aggregates. In reviewing the compressive strengths and absorption tests of the specimens, it was concluded that:

- the strength of the WB with natural fines and coarse aggregates at 90 days was 26MPa (Section 9.3.1) with liquid / solid ratio of 0.42 when compared to the control mix with the same liquid / solid ratio and 100% cement (46MPa) at 90 days of age;
- the ultimate strength (i.e. 90 days of age) of the WB with recycled concrete as fines and coarse aggregates was approximately 23MPa (Section 9.3.3.1) when compared to the control mix using 100% cement with natural fines and coarse aggregates (66MPa) at ultimate strength; and
- the water absorption of the WB with recycled concrete as fines and coarse aggregates at 28 days was 5.6%. The water absorption rate at 10, 30, 60 and 120 minutes cycles was approximately 6, 8, 10 and 12%.

In considering the potential use of the WB with natural fines and coarse aggregates with cement as supplementary material, cement was partially added by four kinds of replacement levels; 5%, 10%, 20% and 30% respectively PG9.5/QWD28.5/GGBS57/PC5, PG9/QWD27/GGBS54/PC10, PG8/QWD24/GGBS48/PC20 and PG7/QWD21/GGBS42/PC30. The results of the laboratory tests concluded that even with 95% cement replacement, highest compressive strengths of approximately 7MPa and 6MPa respectively with 0.42 and 0.50 percent water contents at 7 days were achieved.

10.3 Conclusions Summary

Conclusions summary can be drawn from the follow results:

 the mix with the proportions of 10%PG, 30%QWD and 60%GGBS was identified as the novel mix 'Warwickshire Blend;' achieving compressive strength of up to 41MPa at 180 days of curing.

- refining the preferred mixes to establish the correct liquid / solid ratios was one of the key factors in determining the mixes' optimum performance;
- the hydration process is one of the major factors that influenced the mechanical properties of both the hydraulic binder and sustainable concrete;
- water plays major role in the compressive strength of the sustainable concrete.
 The higher the water content, the lower the compressive strength;
- mixes incorporating V-B5G in the hydraulic pastes require more water to achieve similar workability;
- hydraulic binders incorporating RAF, QWD and GGBS developed strength at extremely slow rate to mixes containing BPD-C, BPD-H and SSD;
- extensive use of the 'Warwickshire Blend' will help contribute the reduction of global CO₂ emissions that come from the manufacturing of cement; and
- the integration of <u>economic</u> regard to the use of little or no cement, <u>environmental</u> in respect to less extraction of natural resources, and <u>social</u> about reducing noise and air pollution due to the close proximity of the by-products source of production, are the three elements of sustainable development FULLY covered in this research study.

CHAPTER 11 – RECOMMENDATIONS FOR FUTURE STUDIES AND APPLICATIONS OF RESEARCH MATERIALS

11.1 Introduction

This chapter focusses on recommendations for future research studies and applications of research materials.

11.2 Recommendations for Future Studies

The following recommendations for future work to be carried out based on the outcome of this research project are:

- it is recommended that the upper limit boundaries used in Minitab 18 for Group 1 (PG80% MHT15% GGBS60%) and Group 5 (V-B5G80% MHT15% SSD60%) and the lower limits for both (PG25% MHT0% GGBS5%) and (V-B5G25% MHT0% SSD25%) respectively can be improved in changing the component percentages to achieve better results;
- it is recommended that further laboratory investigations on durability such as tensile strength, flexural strength, chloride penetration etc. are carried out on the WB sustainable concrete;
- it is recommended that further studies are carried out on the ternary binders to create sustainable concrete from the remaining top two mixes that had potentials: Group 1 (Mix 1 – PG20/RAF20/GGBS60) and Group 2A (Mix 13 – V-B5G10/RAF30/GGBS60);
- it is recommended to understand the mechanism of hydration and chemical reactions of not only the candidate materials but also that of the top two mixes using scanning electron microscopy (SEM); and
- it is recommended that the effect of variability of the by-product materials obtained, even from the same source to be studied closely to achieve consistency in their use and variations.

11.3 Applications of Research Materials

The results of the mixtures obtained from this research study can be used in the following highways and pavement engineering applications:

- mixes with strengths of between 8MPa to 20MPa can be used for base or subbase in accordance with Table 10/1 of (SHW 2016b);
- mixes with strengths of between 0.75MPa to 6.0MPa at 7 days can be used as stabilised base and stabilised sub-base to increase strength and bearing capacity as recommended in the (Road Research Laboratory. Overseas Unit and Great Britain. Overseas Development Administration 1993);
- mixes with low strengths can be used for the protection of existing and new Statutory Undertakers Services apparatus as they could be easily excavated with less energy should the need arise and reinstated in a timely manner; and
- mixes with strengths of up to 20MPa and above have potentials to be used as concrete kerb foundations and backings, foundations for street furniture such as Advanced Directional Signs (ADS), street lighting columns, traffic signal poles and mass concrete slippers to protect damaged parapets subject to the requirements of BS 8500-1.

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APPENDIX A - Questionnaire

Questions to find out about industrial waste minerals that are being disposed of in the UK to landfill sites and their possible applications in road (base) and foundation in pavement engineering

QUESTION		RESPONSE
Q1 (i)	Does your organisation	YES 🗸
	(company) produce industrial	
	waste minerals during the	
	manufacturing process of the	
	main products?	
(ii)	If YES, are the secondary waste minerals produced being taken to landfill sites for disposal?	YES 🗸 NO
(iii)	Are ALL, SOME or NONE of the	ALL
	secondary waste minerals being	SOME 🗸
	produced used in the main	
	manufacturing process of the	NONE
(iv)	If SOME or NONE are used within	YES
	the manufacturing process of the	
	main products, would it be	
	possible for your organisation	
	(company) to send samples to	
	investigate the secondary waste	
	and environmental suitability In	
	(hase) and foundation	
	(Dase) and roundation.	

APPENDIX B – Copy of Correspondence Via Companies Portal

APPENDIX C – Copy of Emails Response from Companies

APPENDIX D – Copy of Emails from Author in Response to Companies Emails.