7/4/05

Eastman Dental Institute (UCL)-Kent-Warwick-Imperial sol-gel project meeting

Ifty Ahmed

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Project Overview:

- The principal aim of the Project is to develop a biomaterial which would prevent orthopaedic implant-associated infections.
- A key new step is developing a sol-gel route to the materials' synthesis which would allow improved compositional and morphological control
  - optimise the sol-gel synthesis routes for these phosphate-based glasses
  - Characterise dissolution properties and profiles of the melt-quenched and sol-gel glasses.
  - Correlate the dissolution properties with the in vitro antibacterial attributes
  - Investigate modern multinuclear solid state NMR techniques, to include O-17 and P-31 data, in order to provide new atomic scale structural information about these materials
  - Apply modern diffraction and X-ray absorption methods to provide new information about the structure of the materials produced
- Ultimately leading to optimised synthesis and processing routes for the generation of materials having a proven ability to kill bacteria and/or prevent adhesion

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My Project:

- To produce the melt-quenched anti-bacterial glasses
- To characterize the melt-quench and sol-gel glasses produced
  - Thermal Analysis
  - XRD Analysis
  - Degradation Analysis
  - Ion Chromatography
- To conduct the microbiological studies on the materials produced

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Copper Glasses:

- Compositions Made

Glass Code	P_2O_5 (mol%)	CaO (mol%)	Na_2O (mol%)	Cu_2O (mol%)	CuO (mol%)	Half Cu_2O (mol%)
0 mol% Cu	45	30	25	0	0	0
1 mol% Cu+1	45	30	24	1	0	0
5 mol% Cu+1	45	30	20	5	0	0
10 mol% Cu+1	45	30	15	10	0	0
1 mol% Cu+2	45	30	24	0	1	0
5 mol% Cu+2	45	30	20	0	5	0
10 mol% Cu+2	45	30	15	0	10	0
1 mol% H Cu+1	45	30	24	0	0	1
5 mol% H Cu+1	45	30	20	0	0	5
10 mol% H Cu+1	45	30	15	0	0	10
Where H = half						

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Characterisation:

- Thermal Analysis
  - Tg, Tc and Tm data obtained.
- Planned Studies:
- XRPD Analysis
- Degradation Studies
- Ion Chromatography
- Currently Underway:
  - Microbiological Studies
  - Zone of Inhibition Studies, and CDFF Studies.

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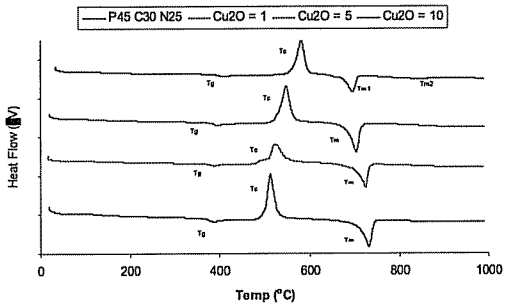
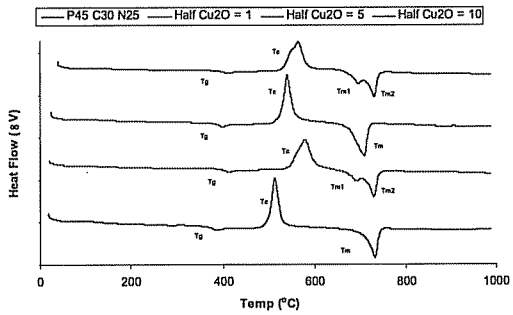
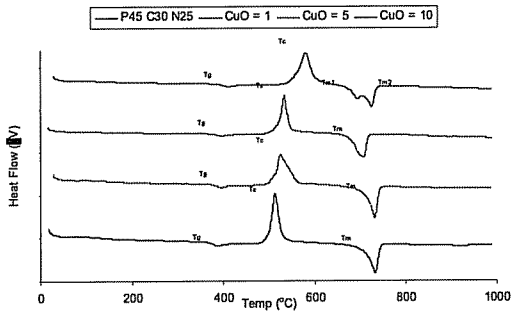
DTA Traces from the Copper Glasses:







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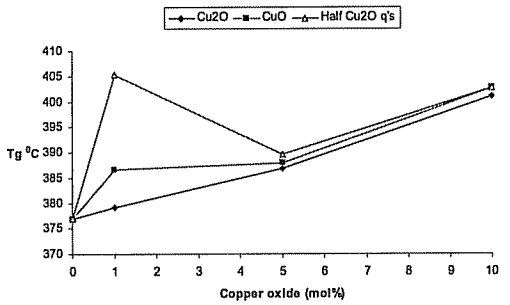
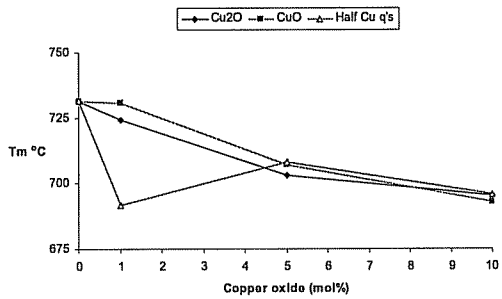
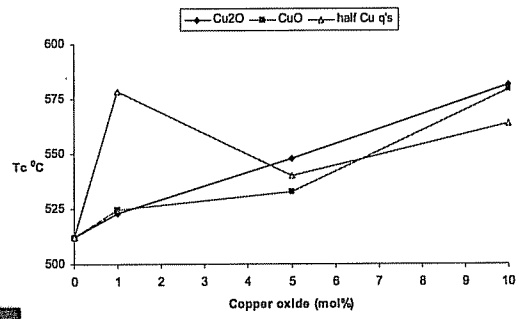
Thermal Parameters of the Copper Glasses:







Silver Glasses:

- Compositions Made

Glass Code	P_2O_5 (mol%)	CaO (mol%)	Na_2O (mol%)	Ag (mol%)
0 mol% Ag	50	30	20	0
5 mol% Ag	50	30	15	5
10 mol% Ag	50	30	10	10
15 mol% Ag	50	30	5	15

Characterisation:

- Thermal Analysis.
- XRPD Analysis
- Degradation analysis
- Ion Chromatography
- Currently Underway:
  - Microbiological Studies
  - Zone of Inhibition Studies, and CDFF Studies.

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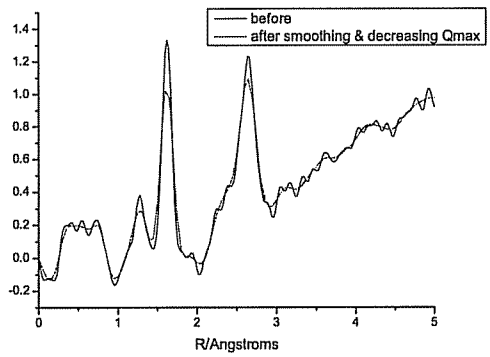
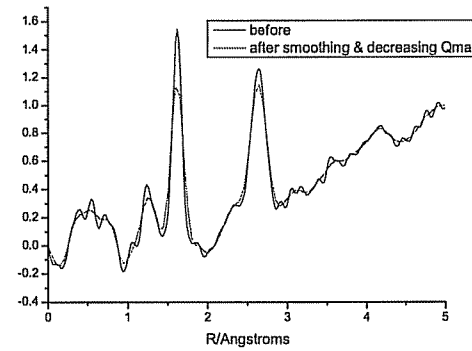
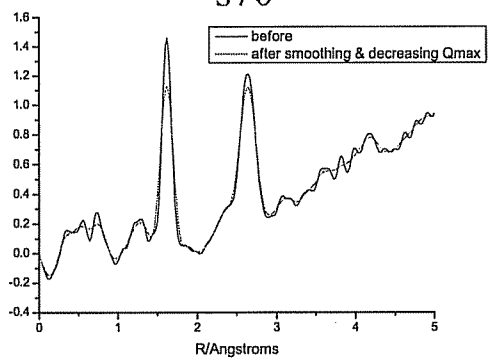
s50



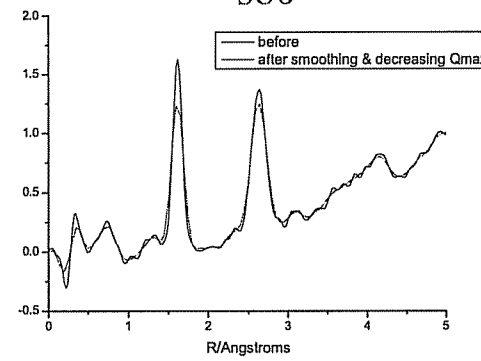
s60



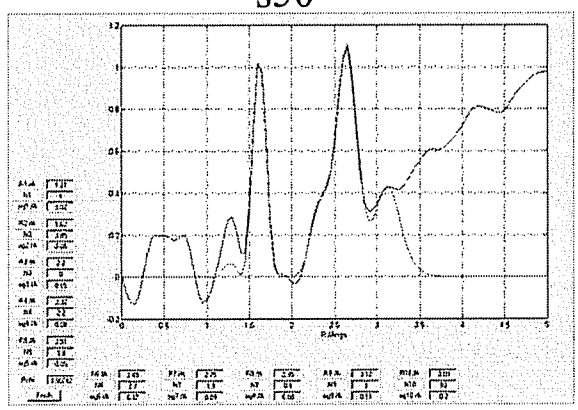
s70



s80

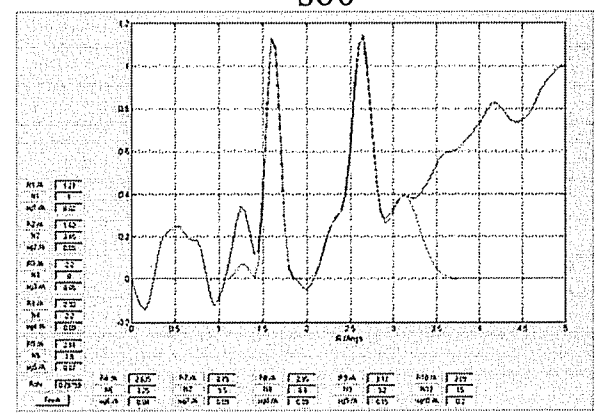


s50



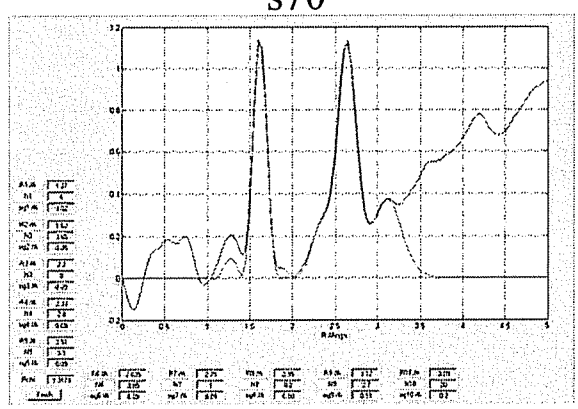
Si-O = 3.85, SCa-O = 6.0, O-O = 2.7

s60



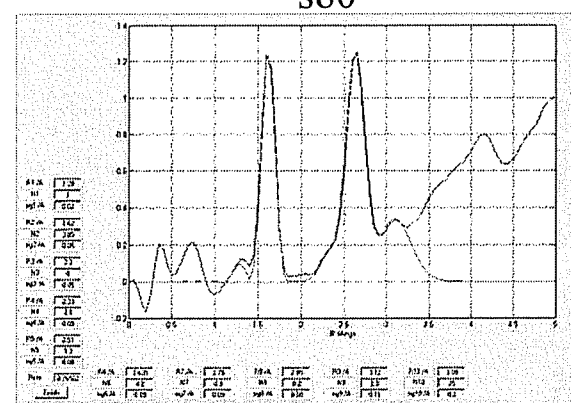
Si-O = 3.65, SCa-O = 5.2, O-O = 3.25

s70



Si-O = 3.65, SCa-O = 4.9, O-O = 3.85

s80

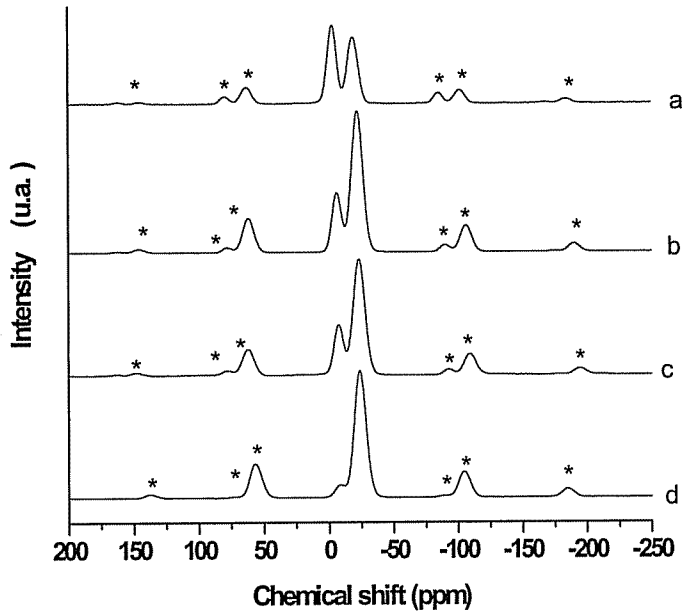


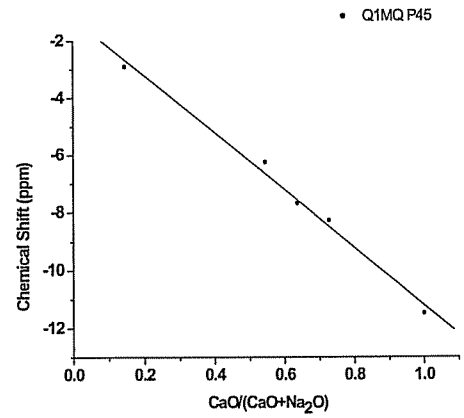
Si-O = 3.85, SCa-O = 5.0, O-O = 4.2

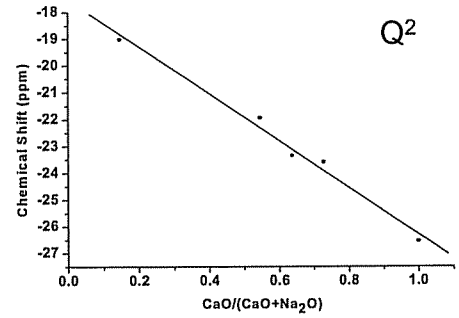
^{31}P MAS NMR

	p(g/cm^3)	Q^0 ppm	Q^1 ppm	Q^2 ppm	Q^3 ppm	Q^0 %	Q^1 %	Q^2 %	Q^3 %
SG-P45C28N7K20	1.53		-8.4	-22.7			15	85	
SG-P45C30N25	1.71		-6.7	-21.9			21	79	
SG-P45C40N15 (untreated)	1.82		-8 ; -10	-23.0			24	76	
SG-P45C40N15 (sonicated)	1.90		-9	-23.0			24	76	
SG-P45C10N20S25	1.28	-0.3	-4.3	-11.2		92	2	6	
SG-P45C20N10S25	1.33	-0.3	-5.2	-11.0		80	12	8	
SG-P45C30S25	1.57	-1.0	-5.0	-12.0		63	14	23	
SG-P45C20N20S15	1.30	0.7	-9.8	-23.0	-31.0	5	15	60	20
SG-P45C30N10S15	1.52	-0.6	-10.9	-24.6	-32.6	10	22	51	17
SG-P45C40S15	1.63	-4.5,-0.9	-11.4	-27.2	-41.3	10	19	63	8
SG-P45C30N15S10	1.37	0.6	-10.0	-23.5	-30.4	2	15	55	28
SG-P45C35N10S10	1.56	-0.3	-10.4	-24.6	-31.8	6	16	60	18
SG-P45C40N5S10	1.60	-0.5	-10.9	-25.1	-32.2	10	25	62	13
MQ-P45C8N47	2.30		-2.9	-19.0			45	55	
MQ-P45C30N25	2.42		-6.1	-22.1			21	79	
MQ-P45C35N20	2.43		-7.2	-23.0			20	80	
MQ-P45C40N15	2.45		-8.1	-23.8			17	83	
MQ-P45C28N7K20	2.10		-7.7	-22.5			20	80	

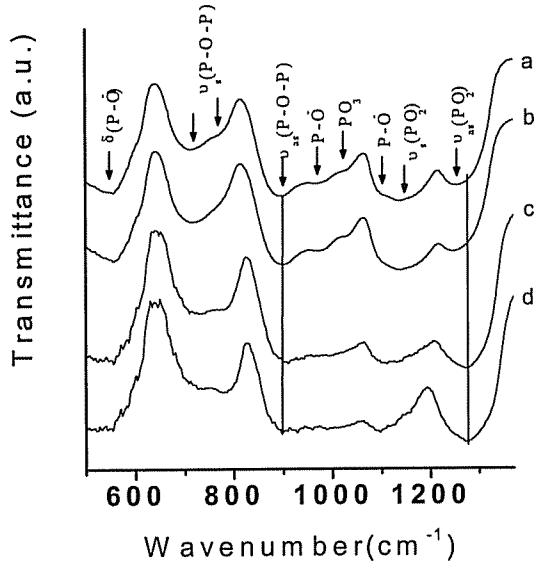
^{31}P MAS NMR

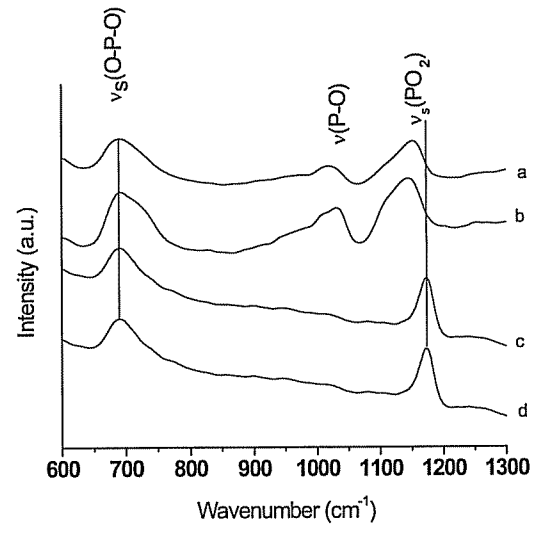






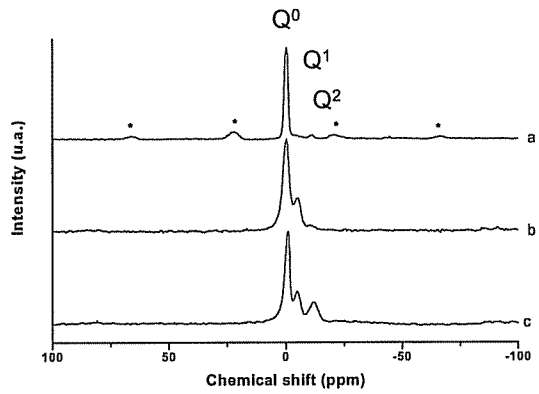
Melt-quenched: P45C8N47 (a), P45C12N43 (b), P45C35N20 (c), P45C40N15 (d)



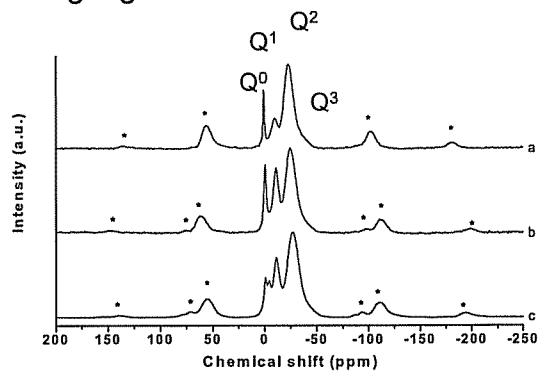


Melt-quenched glasses: a P45C8N47, b P45C12N43, c P45C35N20, d P45C40N15

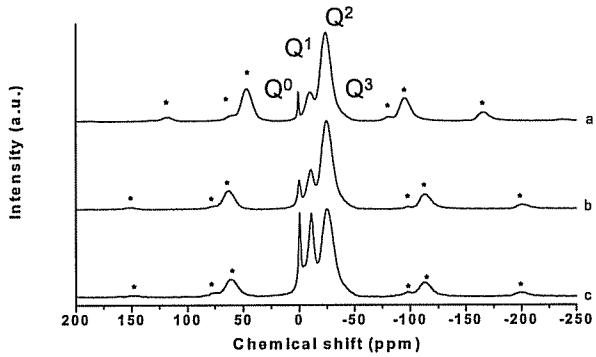
^{31}P MAS NMR - Sol-gel glasses



Sol-gel: P45C10N20S25 (a), P45C20N10S25 (b), P45C30S25



Sol-gel: P45C20N20S15 (a), P45C30N10S15 (b), P45C40S15 (c)



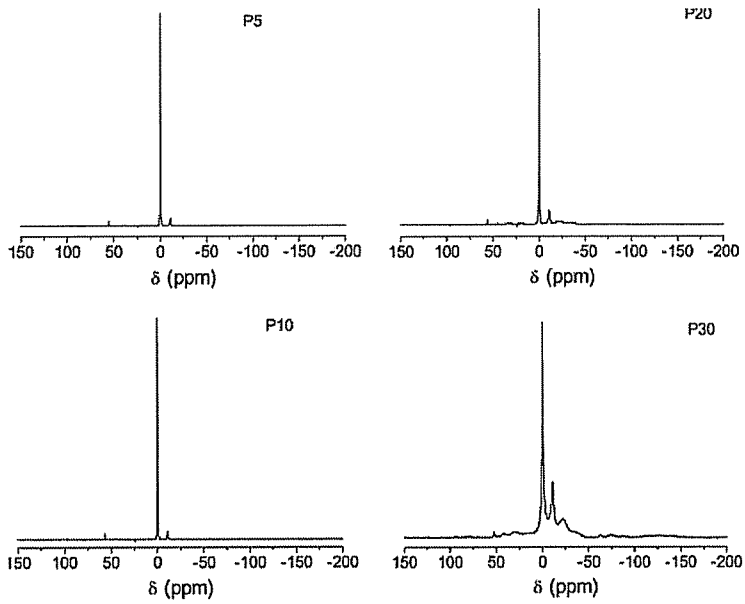
^{29}Si MAS NMR

Resonance at -115 ppm
Si-O-P bonding in

$Si(OSi)_3(OP)$

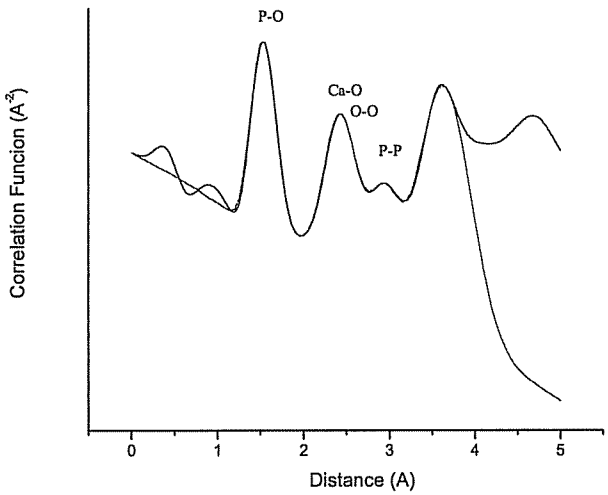
Sol-gel: P45C30N15S10 (a), P45C35N10S10 (b), P45C40N5S10 (c) ^{29}Si MAS NMR spectra of P45C20N10S25 (a), P45C30S25 (b)

Phosphate sol-gel 400C $POCl_3$ $Si(OEt)_4$



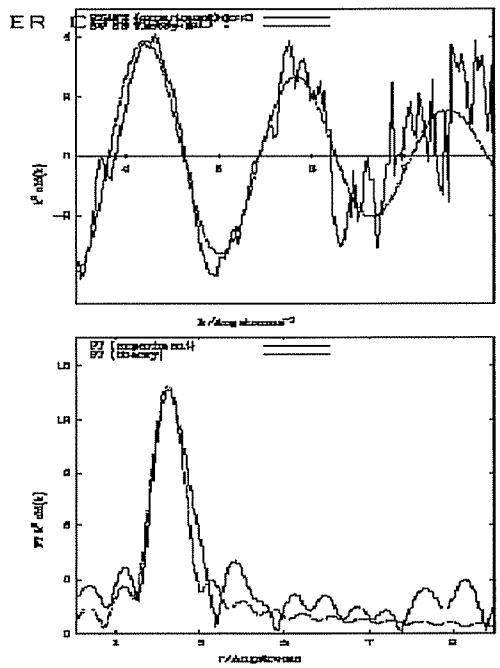
Clayden et al., Journal of Non-Crystalline Solids , Volume 351, Issue 3 , 1 February 2005, Pages 195-202

MQ P50C50



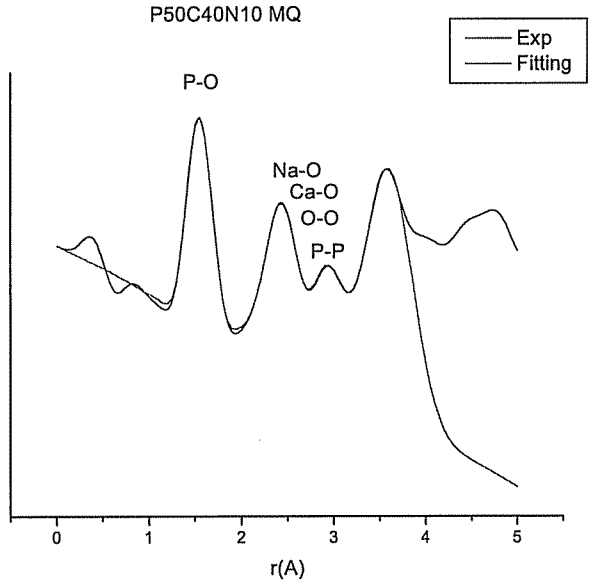
	$P\text{-}O_T$	$P\text{-}O_B$	Ca-O	O-O	P-P
R (A)	1.60	1.49	2.41	2.60	2.92
CN	2.10	2.25	6.65	2.05	3.55

Ca EXAFS

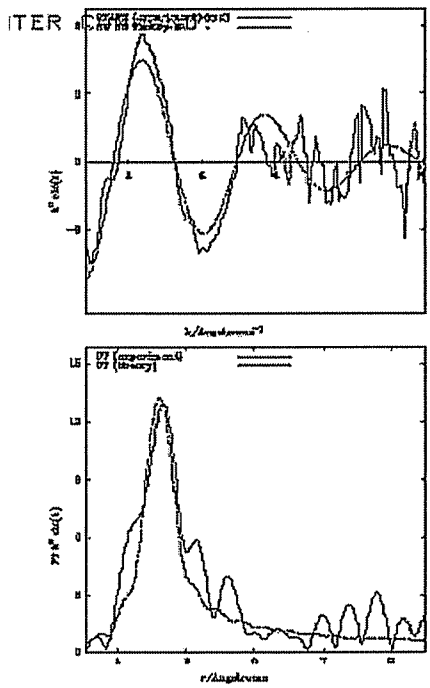


Ca-O
r 2.29
CN 4.4

P50C40N10 MQ

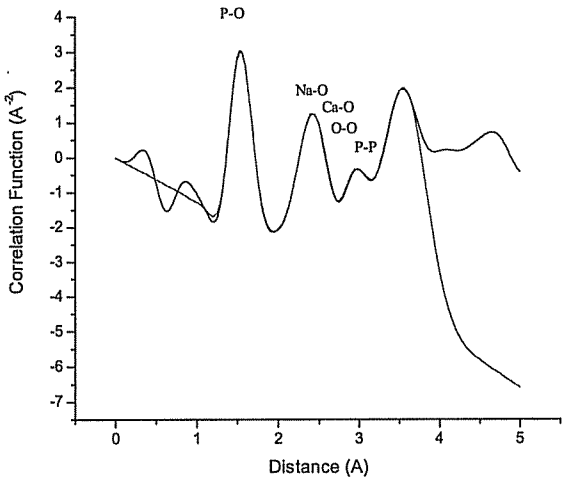


	$P\text{-}O_T$	$P\text{-}O_B$	Na-O	Ca-O	O-O	P-P
R (A)	1.50	1.60	2.43	2.40	2.53	2.92
CN	2.20	2.10	2.00	5.90	2.00	4.90

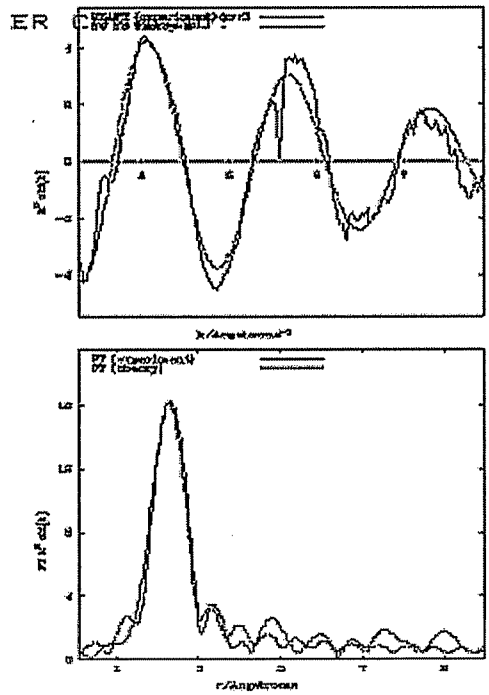


Ca-O
r 2.34
CN 5.0

P45C30N25 MQ

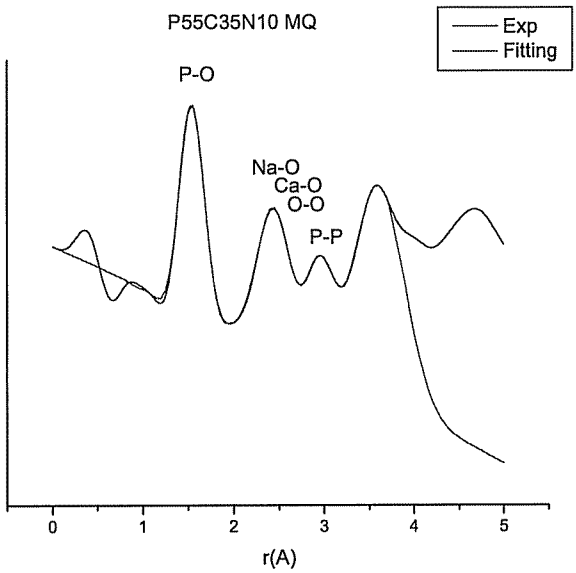


	$P\text{-}O_T$	$P\text{-}O_B$	Na-O	Ca-O	O-O	P-P
R (A)	1.60	1.52	2.35	2.38	2.53	2.94
CN	2.00	2.30	2.20	6.10	4.10	4.50

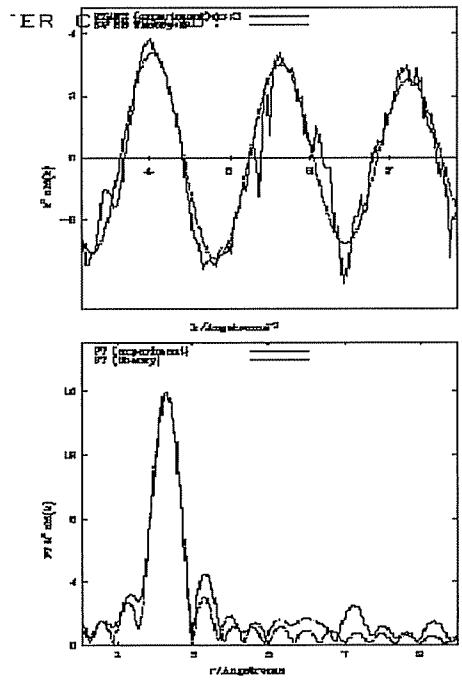


Ca-O
r 2.33
CN 4.9

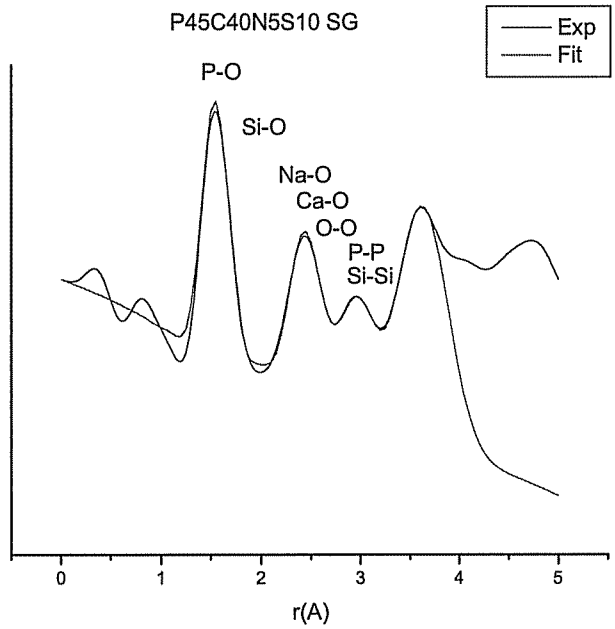
P55C35N10 MQ

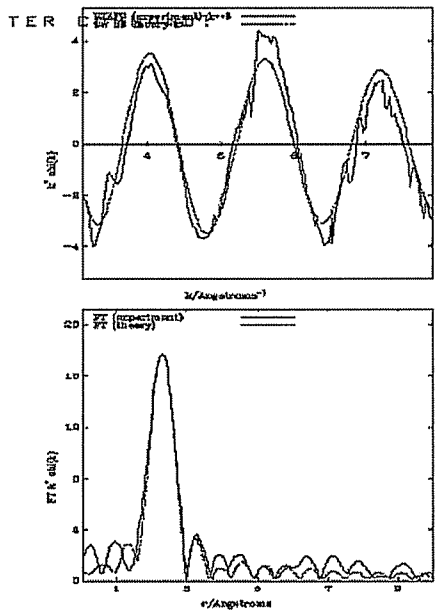


	$P\text{-}O_T$	$P\text{-}O_B$	Na-O	Ca-O	O-O	P-P
R (A)	1.53	1.56	2.46	2.42	2.68	2.97
CN	2.20	2.10	4.00	6.00	4.00	2.20



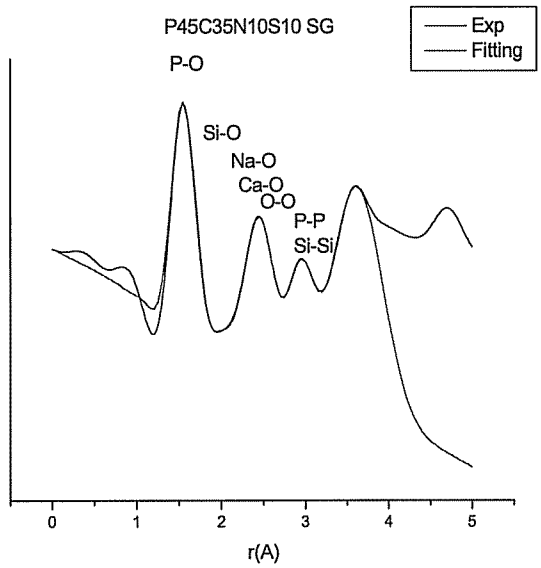
Ca-O
r 2.34
CN 3.2

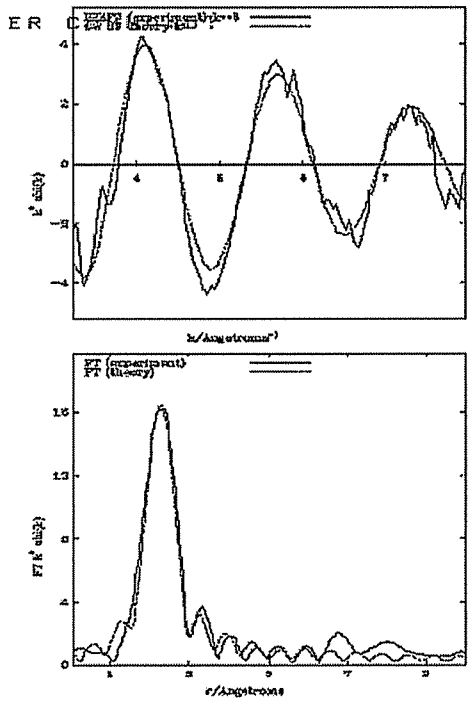




	P-OT	P-OB	Si-O	Na-O	Ca-O	O-O	P-P	Si-Si
R (A)	1.55	1.52	1.61	2.43	2.48	2.70	2.98	3.02
CN	2.00	1.80	4.00	6.00	3.00	2.90	2.30	4.00

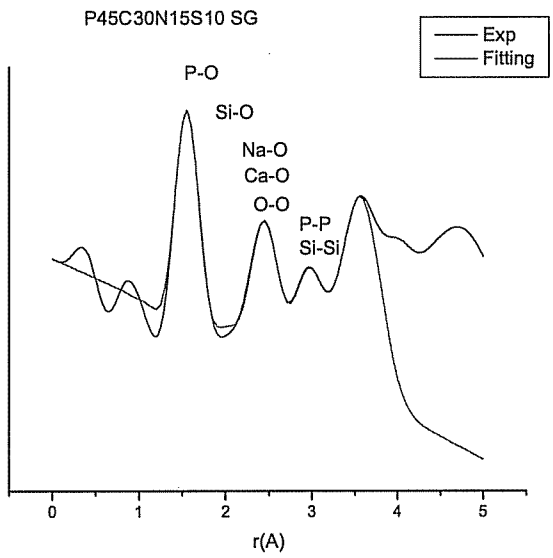
Ca-O
r 2.36
CN 3.2

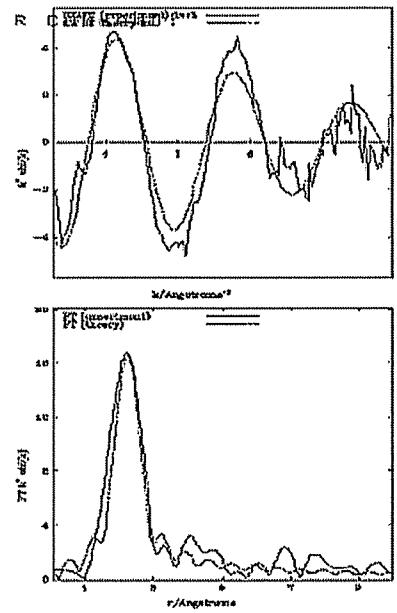




	P-OT	P-OB	Si-O	Na-O	Ca-O	O-O	P-P	Si-Si
R (A)	1.54	1.52	1.61	2.37	2.39	2.56	2.94	3.30
CN	1.30	1.00	4.00	2.20	3.20	3.00	2.30	4.00

Ca-O
r 2.33
CN 4.4





	P-OT	P-OB	Si-O	Na-O	Ca-O	O-O	P-P	Si-Si
R (A)	1.55	1.52	1.61	2.45	2.48	2.70	2.70	2.96
CN	1.90	1.30	4.00	5.70	4.00	1.80	1.80	2.80

Ca-O
r 2.31
CN 5.2

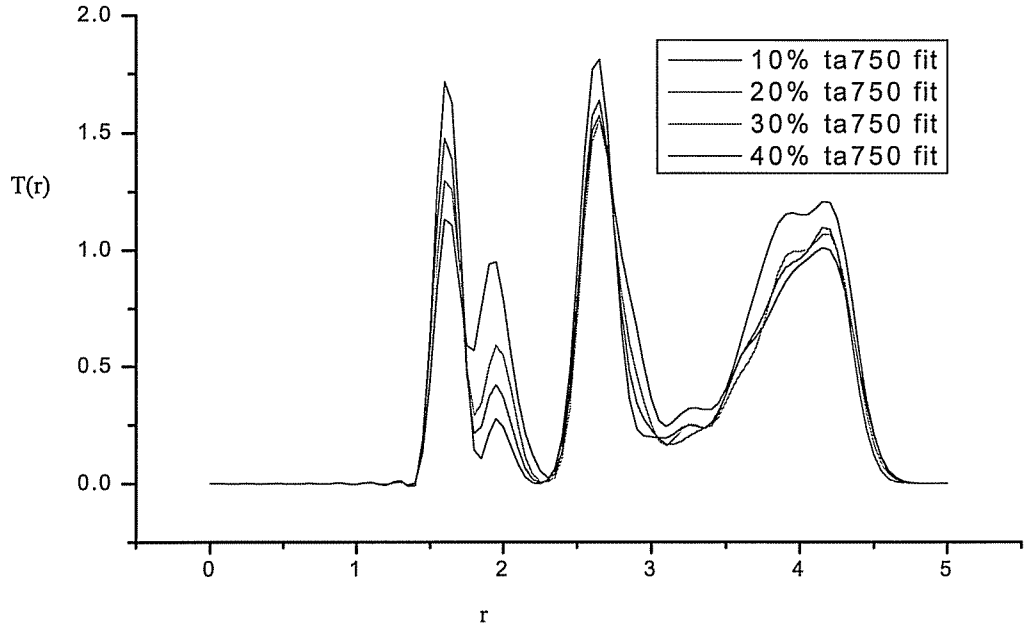
Samples SRS –XRD- Station 9.1

	$P\text{-}O_B$	$P\text{-}O_T$	Si-O	Na-O	Ca-O XRD	Ca-O EXAFS	O-O	P-P	Si-Si
P45C40N15 MQ	1.54 2.5 0.015	1.6 2.5 0.12	-	2.36 3 0.2	2.39 6.2 0.13		2.53 4 0.08	2.93 4.7 0.12	-
P45C30N25 MQ	1.52 2.3 0.003	1.6 2 0.08	-	2.35 2.2 0.11	2.38 6.1 0.12	2.33 4.9	2.53 4.1 0.08	2.94 4.5 0.13	-
P50C50 MQ	1.49 2.25 0.035	1.6 2.1 0.07	-	-	2.41 6.65 0.135	2.29 4.4	2.6 2.05 0.09	2.92 3.55 0.12	-
P50C40N10 MQ	1.5 2.2 0.0012	1.6 2.1 0.0012	-	2.43 2 0.01	2.40 5.9 0.12	2.34 5.0	2.53 2 0.03	2.92 4.9 0.14	-
P55C35N10 MQ	1.53 2.2 0.06	1.56 2.1 0.06	-	2.46 4 0.13	2.42 6 0.12	2.34 3.2	2.68 4 0.19	2.97 2.2 0.04	-
P45C40N5S10 SG	1.55 2 0.08	1.52 1.8 0.025	1.61 4 0.01	2.43 6 0.11	2.48 3 0.01	2.36 3.2	2.7 2.9 0.12	2.98 2.3 0.09	3.02 4 0.09
P45C35N10S10 SG	1.54 1.3 0.05	1.52 1.0 0.05	1.61 4 0.05	2.37 2.2 0.06	2.39 3.2 0.01	2.33 4.4	2.56 3 0.05	2.94 2.3 0.09	3.3 1.4 0.18
P45C30N15S10 SG	1.55 1.9 0.05	1.52 1.3 0.05	1.61 4 0.05	2.45 5.7 0.1	2.48 4 0.12	2.31 5.2	2.7 1.8 0.4	2.7 1.8 0.4	2.96 2.8 0.13

Group Meeting

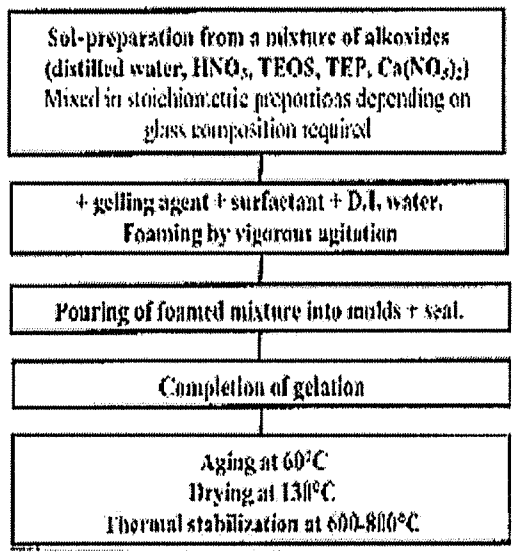
- Neutron data – fits in with XRD but shows they are different samples, RMC being done
- SAXS – resubmit original proposal, also propose experiment on foam synthesis
- 9.1 experiment – XRD on Julian's most bioactive foam

Neutron Data



SAXS

- **New cell 2mm thick to wedge a block of foam in. Has been built and is being tested**
- **Longer camera length**
- **Also propose experiment for synthesis of foams**



Diffraction on 9.1

- Diffraction experiment in June on 9.1
- Julian's foam reacted with SBF between 1 minute and ~ 72 hours
- Maybe some of Ahmad's samples as well