# COLLOIDAL AND CHEMICAL PROPERTIES OF GRAPHENE OXIDE AND STEP WISELY REDUCED GRAPHENE OXIDE

Samar Azizighannad



#### Carbon based materials

- Graphite
- Graphene and Graphene based material-
- Carbon nanotube (single wall and multiwall)
- Fullerene (C60 & C540)
- Diamond
- Amorphous carbon

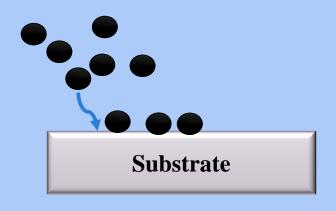
- High surface area
- High electronic Conductivity
- High young's modulus and exceptional mechanical properties
- High thermal conductivity
- High optical transmittance
- Its potential for wide range applications



#### Synthesis of Graphene

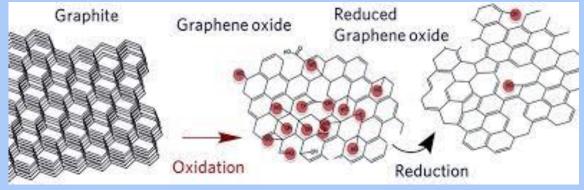


Bottom Up-Chemical Vapor Deposition



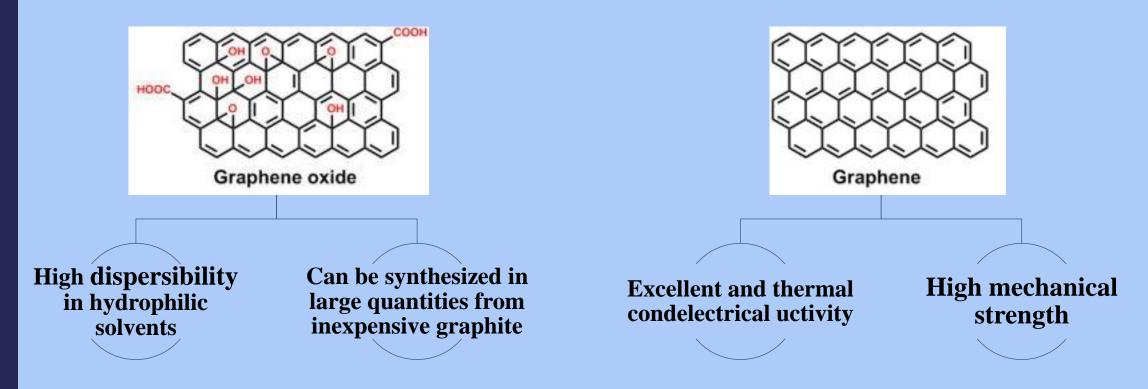


Top Down-Exfoliation of Graphite Oxide



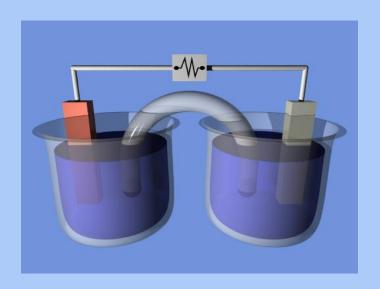


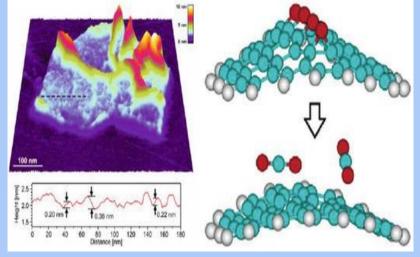
#### Why Controlled reduction important?

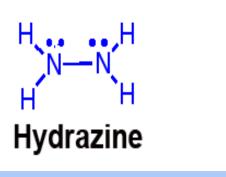


• There is a gap in the literature on the direct comparison on properties of GO and different oxygen level in r-GOs.

#### Synthesis of Reduced graphene Oxide







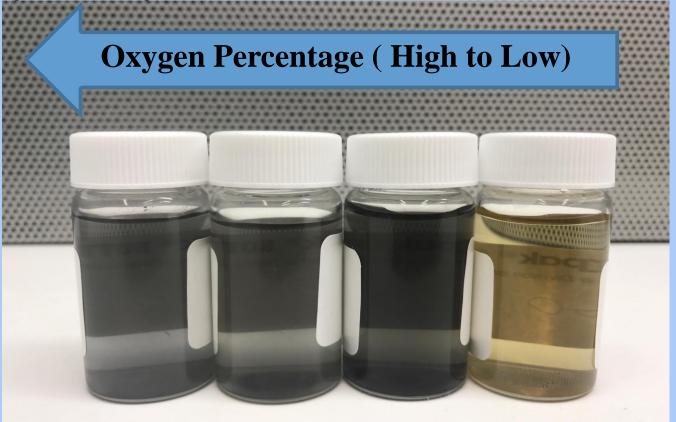
**Electrochemical Reduction** 

Thermal Reduction

**Chemical Reduction** 



# Stepwise Reduction of GO by Metal(Zinc)/Acid Chemical Reduction

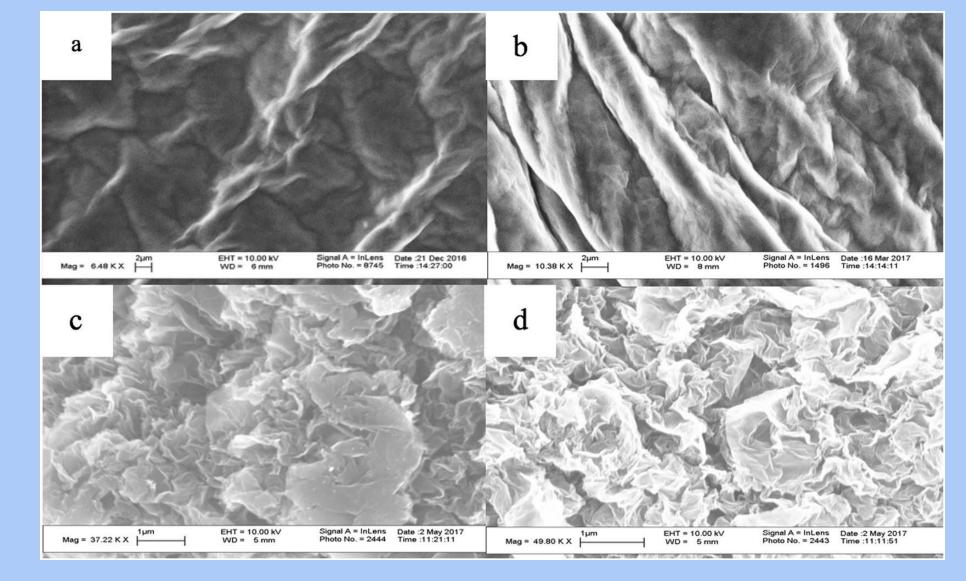




$$Zn \longrightarrow Zn^{2+} + 2e^{-}$$
 and  $GO + aH^{+} + be^{-} \longrightarrow rGO \cdot cH_{2}O$ 

#### SEM (Scanning Electron Microscope)

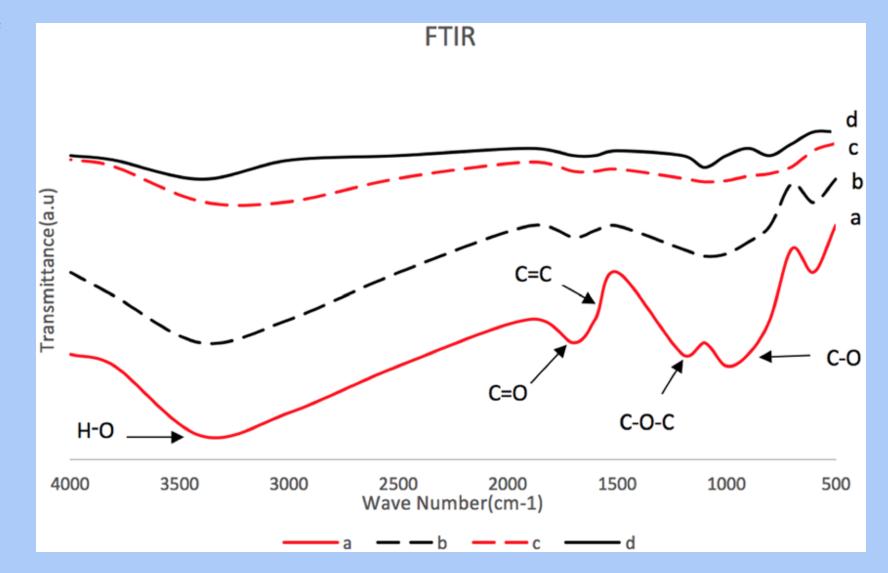
- a) Graphene Oxide
- b) r-GO-31
- c) r-GO-19
- d) r-GO-9





#### FTIR (Fourier Transform Infrared Spectroscopy)

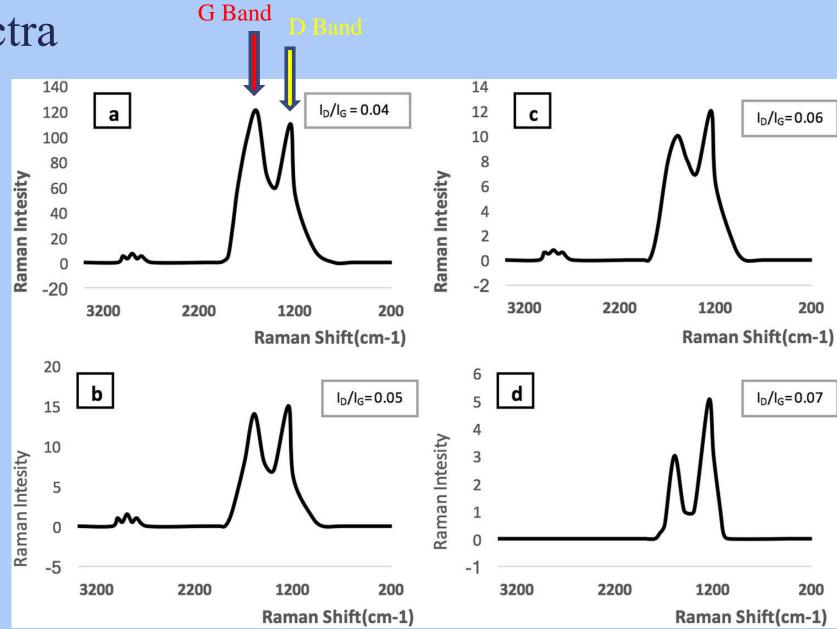
- a) Graphene Oxide
- b) r-GO-31
- c) r-GO-19
- d) r-GO-9





Raman Spectra

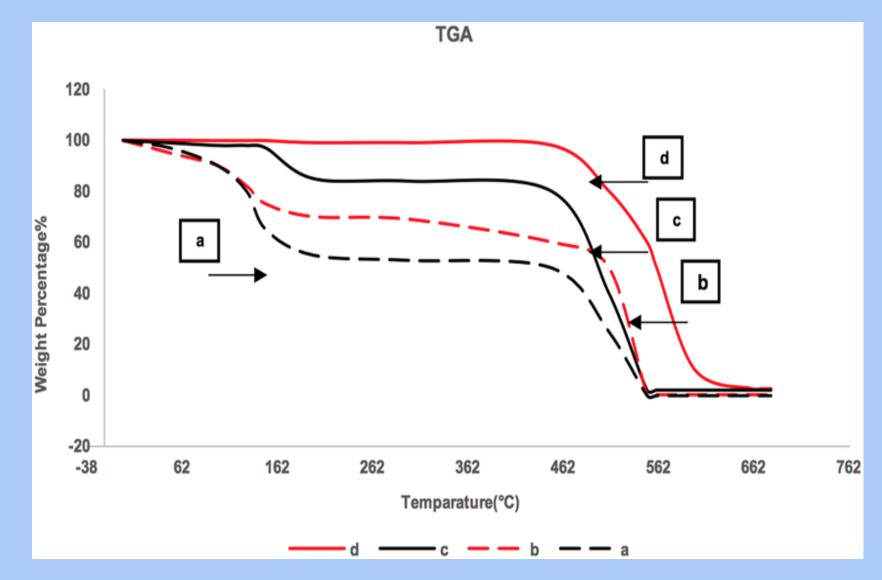
- a) Graphene Oxide
- b) r-GO-31
- c) r-GO-19
- d) r-GO-9





### TGA (Thermogravimetric Analysis)

- a) Graphene Oxide
- b) r-GO-31
- c) r-GO-19
- d) r-GO-9

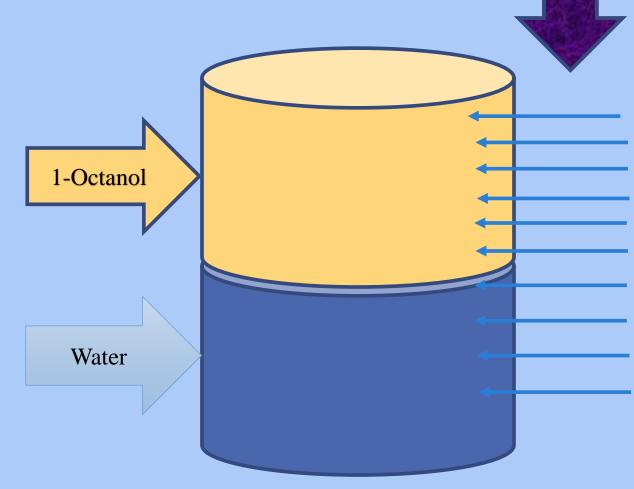




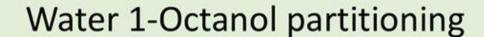
Hydrophobicity Index (HI)

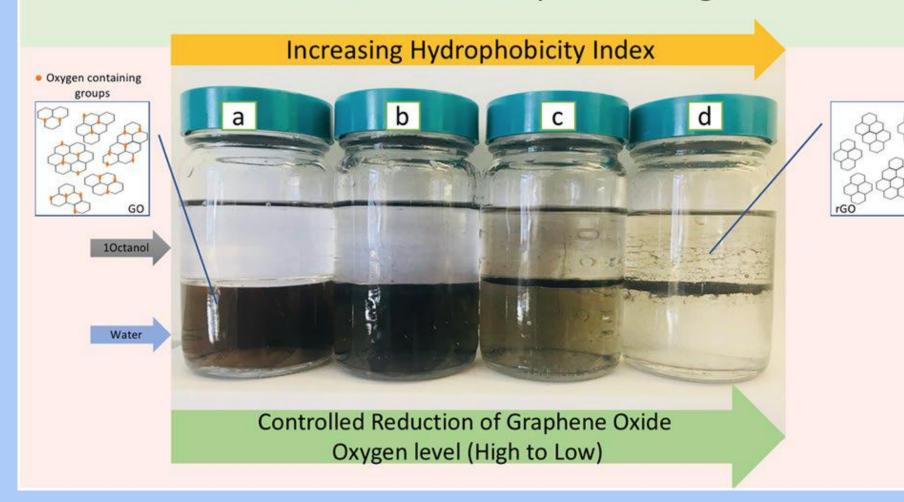
- Dispersibility of GO &rGO in Water
- UV absorbance of GO
  and r-GOs solutions at
  252 nm in water prior to
  and following 1-octanol
  extraction.

$$HI(\%) = \frac{(A_o - A_i)}{A_o} * 100$$



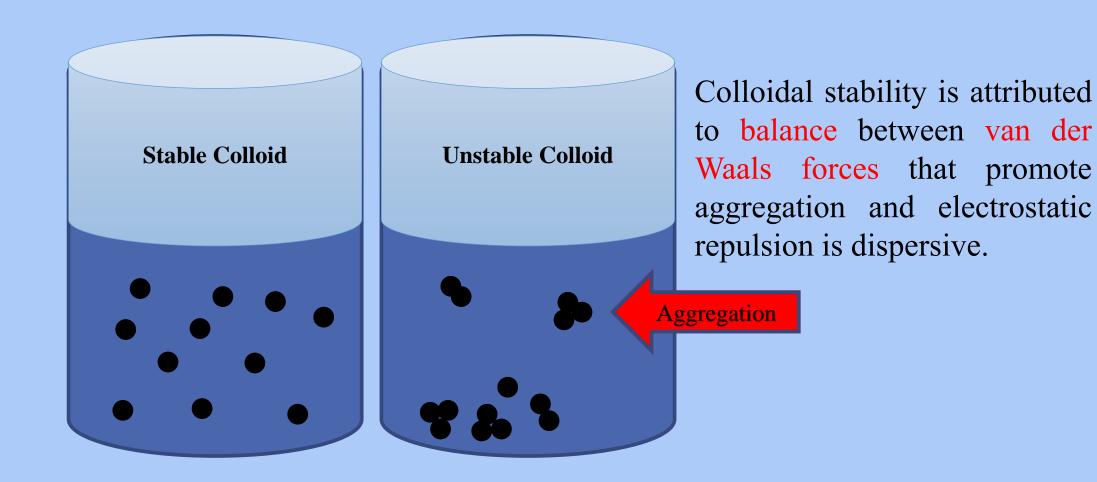
- a) Graphene Oxide
- b) r-GO-31
- c) r-GO-19
- d) r-GO-9





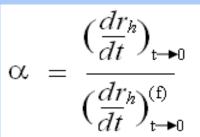


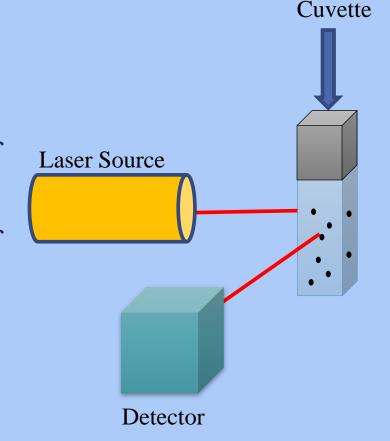
### Colloidal Stability



#### CCC (Critical Coagulation Concentration)

- The aggregation kinetics of the GO and r-GO were studied using time resolved Dynamic Light Scattering
- Particle size regime is faster at lower concentration of salt and it gets fast as salt concentration increases.
- The attachment efficiency α which is the reciprocal of stability ratio of a dispersion were computed Nacl and Mgcl2.



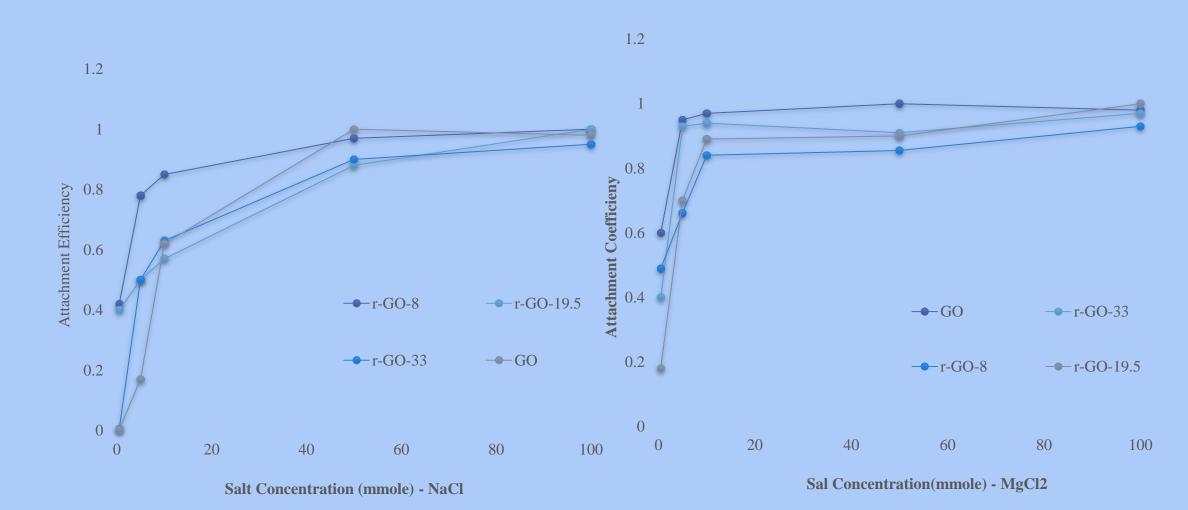


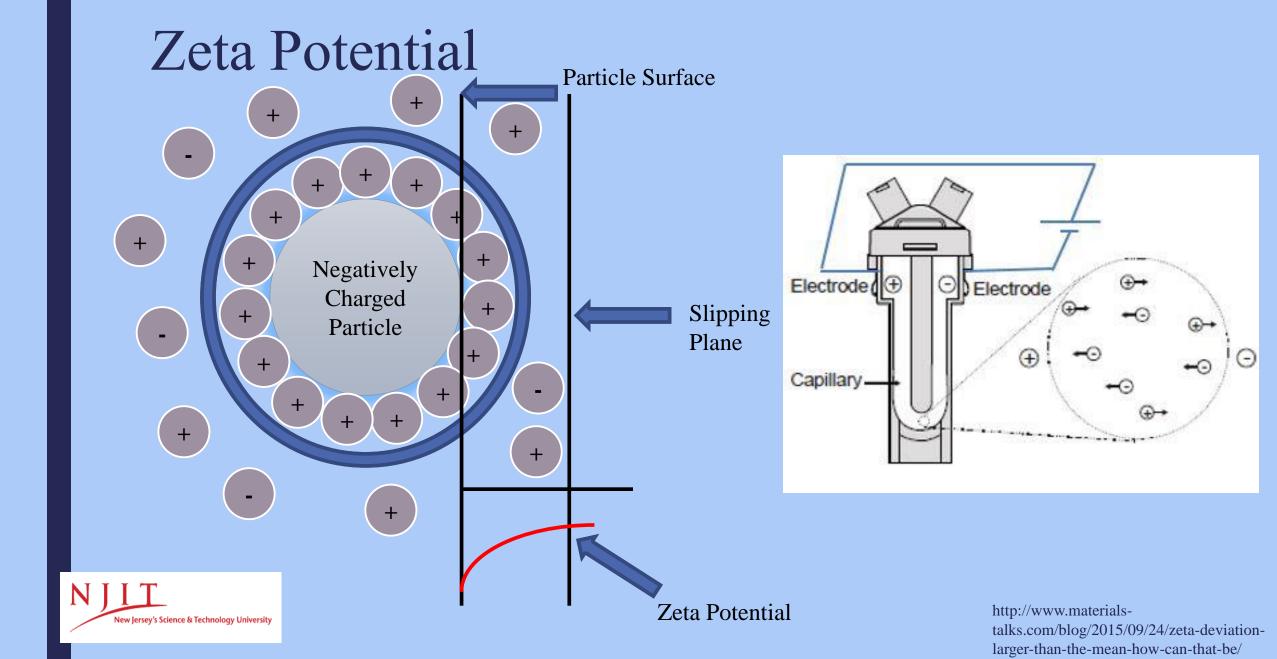
Sample

Containin

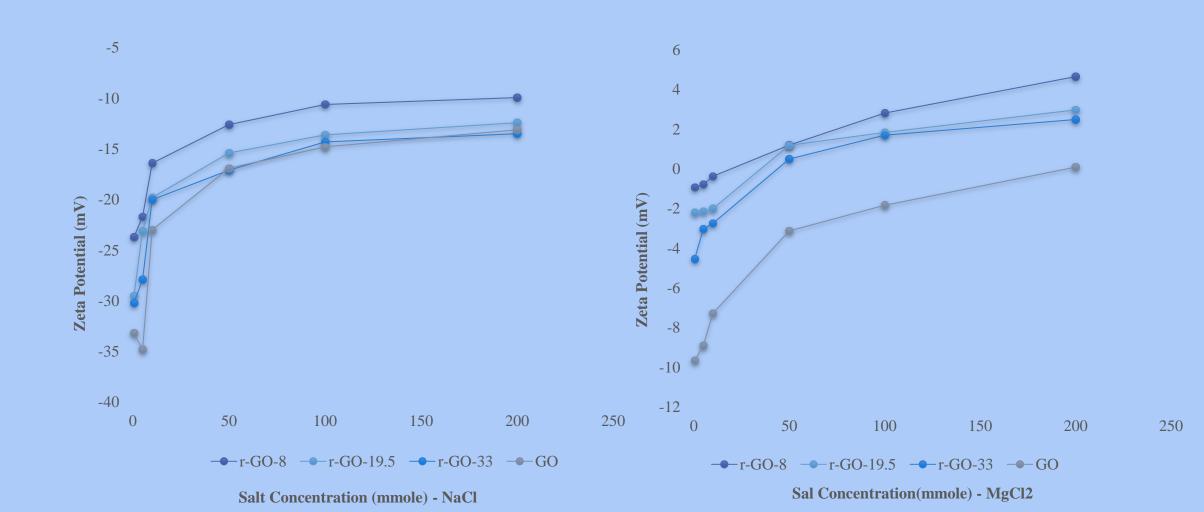


# Attachments Efficiency VS. Salt Concentration





#### Zeta Potential VS. Salt Concentration



## Summery of findings

Analysis/Sample	GO	r-GO-31	r-GO-19	r-GO-9
Percent Carbon	47.48%	66.87%	80.06%	87.71%
Percent Oxygen	49%	31.67%	19.11%	9.69%
$L_a$	22.6	18.5	16	13.4
Particle size in 0.5 mmole/1 NaCl(nm)	642.3	385.5	376.7	327.9
CCC in NaCl	28	27	20	15
Particle size in 0.5 mmole/1 MgCl <sub>2</sub> (nm)	1274	608.2	551.1	358.3
CCC in MgCl <sub>2</sub>	6	6	5	2
Zeta potential in 0.5 mmole/1 NaCl	-33.2	-30.02	-29.5	-23.7
Zeta potential in 0.5 mmole/1 MgCl <sub>2</sub>	-9.66	-4.54	-2.2	-0.92
Hydrophobicity Index	-3.89%	0.98%	1.75%	5.2%
Solubility(µg/ml)	7.4	2.1	~0	~0
Dispersibility(µg/ml)	8	6.3	4.1	2.5



#### Conclusions

- ✓ Stepwise Reduction of Graphene Oxide carried out by Metal/Acid reaction.
- ✓ Zeta potential, particle size distribution and aggregation kinetics were used to study dispersibility of the different r-GO.
- ✓ The GO and r-GO particles began to aggregate with increase in ionic strength.
- ✓ Surface oxidation in r-Go clearly played an important role and higher oxygen content led to higher CCC values.
- ✓ Solubility of graphene oxide in water decreases by decreasing oxygen containing groups.



#### Acknowledgments

- Dr. Somenath Mitra
- Department of Materials Science and Engineering
- NIH (NIEHS)
- Dr. Larisa Krishtopa, Dr. Xueyan Zhang and Dr. Shim
- Research group members: Dr. Wang, Dr. Roy, Worawit, Emine, Madihah, Oindrila and Indrani





## Thank you for Listening

Questions and Comments

