

CHAPTER 3

RESEARCH METHODOLOGY

3.0 INTRODUCTION

The present chapter includes the materials, apparatus, and equipment used for the catalyst synthesis, characterisation and catalytic activity testing throughout the study of the esterification of the pure and dilute acrylic acid with butanol. All the experimental procedures are also delineated.

3.1 MATERIALS

The chemicals used in the present study are listed in the Table 3.1 with its brand, purity, and functions. All these chemicals were used without further purification.

Table 3.1: List of chemical

Chemicals	Assay (%)	Supplier	Function
1-butanol	99.8	Sigma Aldrich	As a reactant
Acrylic acid	99	Aldrich	As a reactant
Amberlyst 15	NA*	Sigma Aldrich	catalyst
Ammonium molybdate tetrahydrate	81-83	Merck	As a reactant for catalyst synthesis
Argentums nitrate	99.8	R&M chemicals	As an indicator
Barium chloride	100.02	Fisher	As an indicator
Butyl acrylate	≥99.5(GC)	Fluka	As a standard for GC-FID analysis
Compressed Air	99.99	Air product	To initiate flame in FID
Helium gas	99.99	Air product	As a mobile phase and carrier for GC-FID analysis
Hydrochloric acid	37	Merck	As a reactant for catalyst synthesis
Hydrogen gas	99.99	Air product	As an inert gas for GC-FID
n-hexane	98	Merck	As a solvent for GC-FID analysis
Nitric acid	65	R&M chemicals	As a reactant for catalyst synthesis
Nitrogen gas	99.99	Air product	As a makeup for GC-FID
Phenolphthalein	NA*	Emory Laboratory Chemicals	As an indicator
Potassium hydrogen phthalate	99.5	Merck	For standardise KOH solution
Potassium hydroxide	85	Merck	For IEC analysis
Sodium chloride	100.1	Fisher	For IEC analysis
Sodium dihydrogen phosphate	99	System	As a reactant for catalyst synthesis
Sodium hydroxide	99	R&M chemicals	As a reactant for catalyst synthesis
Starch		Sigma Aldrich	As a reactant for catalyst synthesis
Sulphated zirconia	NA*	NA*	Catalyst
Sulphuric acid	96	Fisher	As a reactant for catalyst synthesis
Waste expanded polystyrene	NA*	NA*	As a reactant for catalyst synthesis
Zirconium hydroxide	97	Aldrich	As a reactant for catalyst synthesis
Zirconyl chloride octahydrate	98	Sigma Aldrich	As a reactant for catalyst synthesis
ZSM-5 zeolite	NA*	Alfa Aesar	catalyst

*NA denotes not available

3.2 APPARATUS AND EQUIPMENT

3.2.1 Catalyst Characterisation

The sulfonated expanded polystyrene (SEP) were examined using Scanning Electron Microscopy (SEM), thermogravimetric analyser, Fourier Transform Infrared Spectroscopy (FT-IR) and gas pycnometer for its morphology, thermal stability, functional groups and density.

3.2.2 Esterification Reaction Studies

The catalysts were tested through esterification reaction carried out in a three necked flask equipped with condenser, temperature controller and temperature probe. Figure 3.1 shows the experimental setup and Table 3.2 shows the function of each part in the set up.