OPTIMIZATION OF PROCESS PARAMETERS FOR MASTERBATCH SULFUR DISPERSION IN CHEMICAL PLANT

BY

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ABSTRACT

One of the main factors limiting the production of a stable sulfur dispersion in masterbatch or in mass scale is finding the optimized parameters set for each of the processes employed, subject to its particle size, viscosity, pH and TSC. Instable sulfur could affect the final product's quality, for example, "sulfur bloom" on the produced rubber film. In this study, the experimental design follows Taguchi's L₉ orthogonal array to determine the optimum parameters for sulfur dispersion prepared via masterbatch mixing in 4 MT mixing tank; to determine the optimum parameters for sulfur dispersion grinded in 60-Litre grinding mill; and finally, to analyze the morphology of sulfur dispersed in rubber film from selected sulfur dispersion sets. The study involved mixing of sulfur with water and surfactant of a fixed dosage in 4 MT mixing tank with various mixing speed and mixing time. The sulfur dispersion with optimized mixing parameter set was chosen for the grinding mill optimization which involved various grinding mill's motor speed and pump rate. The plot of means and tabulated ANOVA based on respective S/N ratio show that particle size and viscosity were mostly affected with both processes' parameter changes. At higher mixing speed of 800 rpm and longer mixing time of 30 minutes, an optimized sulfur dispersion was able to be obtained according to the viscosity and particles size responses. It was also found that higher motor speed and lower pump rate of the grinding mill at 800 rpm and 350 L/min respectively produces smaller particle size of sulfur dispersion which also hinders severe formation of sulfur crystals observed on the casted rubber film. Severe sulfur crystals were observed on the rubber film's casted with larger sulfur particle size or 'less stable' sulfur dispersion. Overall, higher mixing speed and longer mixing time, followed by higher grinding mill's motor speed and lower pump rate favors the production of smaller particle size of sulfur dispersion that is optimized and is more stable especially when applied in rubber products.

خلاصة البحث

أحد العوامل الرئيسية التي تحد من إنتاج الكبريت الثابت المنتشر في الماستر باتش أو على نطاق واسع هو العثور على المتغيرات المحسنة المحددة لكل من العمليات المستخدمة، وفقًا لحجم الجسيم واللزوجة ودرجة الحموضة وTSC. يمكن أن يؤثر عدم استقرار الكبريت على جودة المنتج النهائي، على سبيل المثال، دمج الكبريت على الفيلم المطاطي الذي يتم إنتاجه. تطبق هذه الدراسة تصميمًا تجريبيًا يتضمن مصفوفة تاجوشي المتعامدة L9 لتحديد المتغيرات المثلي لانتشار الكبريت والذي سيتم تحضيره من خلال خلط الماستر باتش في خزان خلط سعته 4MT؛ ولتحديد المتغيرات المثلي لانتشار الكبريت الذي تم طحنه في مطحنة تسع 60 لترًا؛ وأخيرًا، لتحليل شكل الكبريت المنتشر في الفيلم المطاطي من مجموعات مختارة من الكبريت المنتشر. تضمنت الدراسة خلط الكبريت بالماء وخافض التوتر السطحي بجرعة ثابتة في خزان خلط 4MT مع سرعة خلط ووقت مختلفين. تم اختيار الكبريت المنتشر ذو المجموعة المثلى من متغيرات الخلط لتحسين الطحن والتي تضمنت سرعات طحن ومعدلات ضخ مختلفة. تُظهر مجموعة الوسائل وANOVA المجدولة بناءً على نسبة S / N ذات الصلة أن حجم الجسيمات واللزوجة تأثرتا بشكل كبير بتغير عوامل العمليتين. عند سرعة خلط عالية (تبلغ 800 دورة في الدقيقة) ووقت خلط أطول (يبلغ 30 دقيقة) يمكن الحصول على انتشار كبريت أمثل وفقًا لاستجابات اللزوجة وحجم الجسيمات. أظهرت النتائج أيضًا أن سرعة المحرك العالية ومعدل الضخ المنخفض للطاحونة واللذان يبلغان 800 دورة في الدقيقة و350 لتر/ دقيقة على التوالي ينتجان حجم جزيئات أصغر للكبريت المنتشر مما يعيق التكوين المكثف لبلورات الكبريت التي تم رؤيتها على فيلم المطاط. تم ملاحظة وجود بلورات كبريتية مكثفة على الفيلم المطاطى بجسيمات كبريت ذات حجم أكبر أو انتشار كبريت "أقل استقرارًا". بشكل عام، فإن سرعة الخلط الأعلى ووقت الخلط الأطول، تليها سرعة الطحن الأعلى ومعدل الضخ المنخفض تتسبب في إنتاج حجم جزيئات أصغر من الكبريت االمنتشر الذي يتم تحسينه ويكون أكثر استقرارًا خاصة عند استخدامه في المنتجات المطاطية.

APPROVAL PAGE

I certify that I have supervised and read this study and that in my opinion, it conforms to acceptable standards of scholarly presentation and is fully adequate, in scope and quality, as a thesis for the degree of Master of Science (Biotechnology Engineering).

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Assalamualaikum w.b.t.,

In the name of Allah, The Most Gracious, The Most Merciful. All glories and praises are to Allah the Almighty, Lord of the Universe, the Merciful and Beneficent to Prophet Muhammad S.A.W, His Companion and the people who follow His path. Allah's Blessings and Mercies on me ease the herculean task of accomplishing this thesis.

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TABLE OF CONTENTS

Abstracti		
Abstract in Arabic		
Approval page		
Declaration		
Copyright Page v		
Ackno	wledgements	viii
List of	Tables	xi
List of	Figures	xiv
List of	Abbreviations	XV
List of	Symbols	xvi
		'
CHAI	PTER 1: INTRODUCTION	1
1.1	Background of Study	1
1.2	Problem Statement	2
1.3	Importance of Study	4
1.4	Objectives	5
1.5	Scopes of Study	5
1.6	Thesis Organization	7
110	Inters organization	····· ′
СНАН	PTER 2: LITERATURE REVIEW	9
2.1	Background of Sulfur	9
	2.1.1 Sulfur Dispersion as Rubber Vulcanizing Agent	11
2.2	Usages of Sulfur Element	14
	2.2.1 Sulfur Dispersion as Rubber Vulcanizing Agent	16
	2.2.2 Issues with Sulfur Dispersion in Rubber and Possible Causes	18
23	Process Ontimization Using Taguchi's Method	20
2.3	2.3.1 Taguchi's Approach	20
	2.3.2 Different Industries Ontimization Study Using Taguchi's Approac	h
	2.0.2 Different maasures optimization stady osing ragioni strippicae	22
	2.3.3 Material Mixing and Grinding Mill Process Optimization Studies	24
2.4	Surfactant Type And Formulation of Sulfur Dispersion	27
2.1	Chapter Summary	34
2.5	Chapter Summary	
СНАЕ	PTER 3: MATERIALS AND METHODS	36
3.1	Flowchart on Optimization Study	36
3.2	Main Materials	38
5.2	3.2.1 Chemicals	38
	3.2.7 Process Equipment/Machinery	39
33	Fxperimental Design: Taguchi's Method	39
5.5	3 3 1 Mixing Process	39
	3.3.2 Grinding Mill Process	<u></u>
3 /	Latex Compounding & Rubber Film Casting	<u></u>
3.4	Characterizations	
5.5	3.5.1 Particle Size Distribution	
	3.5.2 TSC of Sulfur Dispersion	<u>46</u>

	3.5.3	pH Value of Sulfur Dispersion	46
	3.5.4	Viscosity of Sulfur Dispersion	47
	3.5.5	Stability Analysis	48
	3.5.6	Rubber Film Morphology Observation	48
3.6	Chapte	er Summary	49
CHA	PTER 4	: RESULTS & DISCUSSION	50
4.1	Proces	s Optimizations of Sulfur Dispersion in Mixing Tank (Objective	21) 50
	4.1.1	Particle Size Analysis from Sulfur Mixing	51
	4.1.2	Viscosity Analysis from Sulfur Mixing	54
	4.1.3	pH Analysis from Sulfur Mixing	57
	4.1.4	TSC Analysis from Sulfur Mixing	60
4.2	Proces	s Optimizations of Sulfur Dispersion In Grinding Mill (Objectiv	re 2) 63
	4.2.1	Particle Size Analysis from Sulfur Grinding	65
	4.2.2	Viscosity Analysis from Sulfur Grinding	68
	4.2.3	pH Analysis from Sulfur Grinding	71
	4.2.4	TSC Analysis from Sulfur Grinding	74
4.3	Sulfur	Dispersion Stability & Morphology (Objective 3)	77
	4.3.1	Sulfur Dispersion Stability Analysis	78
	4.3.2	Sulfur Morphology in Rubber Film	80
		4.3.2.1 LED Magnifier Lens	80
		4.3.2.2 SEM/EDX Analysis	84
4.4	Chapte	er Summary	
СНА	PTER 5	: CONCLUSION	88
5.1	Conclu	ision	88
5.2	Recom	nmendations	90
DFFI	DENCI	FC	01
	ENDIX A	а А	90
APPF		* R	100
APPF	ENDIX C	с	103
LIST	OF PU	BLICATIONS	106

LIST OF TABLES

Table 2.1	Basic properties of sulfur element	12
Table 2.2	Different forms of sulfur, industrial applications and their usage	15
Table 2.3	Different studies on optimization using Taguchi's approach (S/N ratio and sensitivity analysis)	23
Table 2.4	Optimization studies have been done related to stirring conditions	25
Table 2.5	Optimization studies have been done related to grinding or milling conditions	26
Table 2.6	Examples of formulation for sulfur dispersions	33
Table 3.1	Internal controlled range on sulfur's chemical specification	38
Table 3.2	Ingredients in preparing sulfur dispersion	39
Table 3.3	Controlled factors and its respective levels for sulfur mixing process	40
Table 3.4	DOE of Taguchi's L ₉ Orthogonal Array extended from Table 3.3	40
Table 3.5	Controlled factors and its respective levels for sulfur grinding process	41
Table 3.6	DOE of Taguchi's L ₉ Orthogonal Array extended from Table 3.5	42
Table 3.7	Formulation of Natural Rubber latex added with sulfur of various grinding mill parameter	44
Table 4.1	Experimental results for particle size of sulfur dispersion after mixing	52
Table 4.2	ANOVA: Sulfur dispersion mixing particles size, D90	54
Table 4.3	Experimental results for viscosity of sulfur dispersion after mixing	55
Table 4.4	ANOVA: Sulfur dispersion mixing viscosity	57
Table 4.5	Experimental results for pH of sulfur dispersion after mixing.	59
Table 4.6	ANOVA: Sulfur dispersion mixing pH	60

Table 4.7	Experimental results for TSC of sulfur dispersion after mixing	61
Table 4.8	ANOVA: Sulfur dispersion mixing TSC	62
Table 4.9	Experimental results for particle size of sulfur dispersion after grinding mill	66
Table 4.10	ANOVA: Sulfur dispersion grinding particle size, D90	68
Table 4.11	Experimental results for viscosity of sulfur dispersion after grinding mill	69
Table 4.12	ANOVA: Sulfur dispersion grinding viscosity	71
Table 4.13	Experimental results for pH of sulfur dispersion after grinding mill	73
Table 4.14	ANOVA: Sulfur dispersion grinding pH	74
Table 4.15	Experimental results for TSC of sulfur dispersion after grinding mill	75
Table 4.16	ANOVA: Sulfur dispersion grinding TSC	76
Table 4.17	Visual observation of rubber film with different sulfur dispersion grinding mill parameters using LED magnifier lens	81
Table 4.18	SEM/EDX elemental analysis on rubber film of different sulfur dispersion grinding mill parameters	84
Table A1.1	Project Milestone/Gantt Chart for Full Research Master Study	99
Table A2.1	Sulfur mixing specifications result from first run	100
Table A2.2	Sulfur mixing specifications result from second run	100
Table A2.3	Sulfur mixing specifications result from third run	100
Table A2.4	Experimental results for sulfur dispersion mixing viscosity	101
Table A2.5	Experimental results for sulfur dispersion mixing pH	101
Table A2.6	Experimental results for sulfur dispersion mixing TSC	102
Table A2.7	Experimental results for sulfur dispersion mixing particles size, D90	102
Table A3.1	Sulfur grinding specifications result from first run	103
Table A3.2	Sulfur grinding specifications result from second run	103

Table A3.3	Sulfur grinding specifications result from third run	103
Table A3.4	Experimental results for sulfur dispersion grinding mill viscosity	104
Table A3.5	Experimental results for sulfur dispersion grinding mill pH	104
Table A3.6	Experimental results for sulfur dispersion grinding mill TSC	105
Table A3.7	Experimental results for sulfur dispersion grinding mill particles size, D90	105

LIST OF FIGURES

Figure 2.1	Sulfur production by year and country (2013 until 2018)	10
Figure 2.2	Clauss process used to yield sulfur element	11
Figure 2.3	Dispersion of solids in aqueous system (Tadros, 2013)	17
Figure 2.4	A surfactant micelle in water with three different regions identified: outer region, palisade region and core.	30
Figure 3.1	Flowchart on Sulfur Dispersion Optimization Study	37
Figure 4.1	Effect of mixing parameters on particle size, D90 mean of means	53
Figure 4.2	Illustration on agitation means in solid particles dispersion by breaking particle-particle interactions	53
Figure 4.3	Effect of mixing parameters on viscosity mean of means	54
Figure 4.4	Effect of mixing parameters on pH mean of means	59
Figure 4.5	Effect of mixing parameters on TSC mean of means	62
Figure 4.6	Effect of grinding mill parameters on particles size, D90 mean of means	67
Figure 4.7	Illustration on particles in between rolling beads in solid particles dispersion by grinding mill	67
Figure 4.8	Effect of grinding mill parameters on viscosity mean of means	70
Figure 4.9	Effect of grinding mill parameters on pH mean of means	73
Figure 4.10	Effect of grinding mill parameters on TSC mean of means	76
Figure 4.11	Instability index of different sulfur dispersion grinding parameters	78
Figure 4.12	Mechanism of stable and unstable particles in dispersion	80

LIST OF ABBREVIATIONS

- TSC Total Solids Content
- S Sulfur (element)
- H₂S Hydrogen Sulfide
- SO₂ Sulfur Dioxide
- CMC Critical micelle concentration
- MT (unit) Metric-tonne
- rpm (unit) Revolutions per minute
- cP (unit) centi-Poise
- μm (unit) micro-meter
- L/min (unit) Litre per minute
- Eq. Equation
- S/N ratio Signal-to-noise ratio
- DOE Design of experiments
- ANOVA Analysis of variance
- DF Degree of freedom
- Adj MS Adjusted mean squares
- Adj SS Adjusted sum of squares
- LED Light-emitting diode
- SEM/EDX Scanning Electron Microscopy / Energy Dispersive X-Ray Analyzer

LIST OF SYMBOLS

- + positive charge
- negative charge
- \sum summation
- ° degree
- % percent
- μ micro

CHAPTER 1 INTRODUCTION

1.1 BACKGROUND OF STUDY

Sulfur production over the world is considerably increasing over the past years to date, reflecting its high demand by different industrial applications. The sulfur element may undergo many distinguish processes to yield different forms of sulfur that are deemed suitable for the end-user's respective usage objective.

Additionally, tackling issues with sulfur especially on the sulfur bloom as in rubber vulcanization is crucial in order to maintain the quality of their end-products. Key industries such as rubber has been considerably consuming a large amount of sulfur (in dispersion form) for their production, particularly during their latex compounding procedure (blending of latex, sulfur dispersion, and other additives which are typically in dispersion form). Promising studies such as optimizing sulfur dispersion's manufacturing parameters are possible to be done so as to highly utilize the exceptionally precious sulfur without wastages (Omar, Ali, Mohamad, & Azmi, 2019).

The Taguchi approach is greatly used in any manufacturing unit or industries to determine the optimal combination of process' parameters and responses for better performance (Chen, Li, & Cox, 2009). Since this approach offers a great focus on process enhancement strategy on continuous improvement and innovation, it is best employed for the improvement of existing technology of any industries (Jirasukprasert, Garza-Reyes, Kumar, & Lim, 2015). Additionally, Taguchi's parameter design is an experiment-based process that covers the following scopes or steps to identify settings of design parameters that optimizes performance characteristics or response(s):

- i. Identify initial and competing settings of design parameters, as well as important noise factors and their ranges.
- ii. Construct the design and noise matrices, and plan the parameter design experiments.
- iii. Conduct the parameter design experiments and evaluate the performance statistic for each test run of the design matrix.
- iv. Use the values of the performance statistic to predict new settings of the design matrix.

Thus, this study proposed the empirical application of Taguchi's approach to optimize the process steps that in turn can improve quality of sulfur dispersion product in a chemical dispersion manufacturing plant in Malaysia. The DOE and response analysis for the process validation will be done with Taguchi's DOE and ANOVA.

1.2 PROBLEM STATEMENT

Producing high quality sulfur dispersion is a challenging process for chemical manufacturers, where a lot of rubber goods performance mostly depends on sulfur dispersion's quality characteristics' compliance. Sulfur is usually prepared as colloid or dispersion state via mixing and/or chemical grinding process that will produce fine sulfur particle size that rarely settles or sediments. This sulfur dispersion in turn will be suitable to be used in latex compounds. Several studies have been done with

different sulfur types and optimal ball-milling process conditions that is proven could comparatively give obvious changes in rubber vulcanizate's mechanical properties (Pangamol et al., 2016b). Thus, it is suggested that improper optimization of process conditions such as ball-milling to effectively reduce the particle size and/or their distributional behavior could remarkably affect its stability or dispersibility in the rubber matrix.

Quality issue often associated with sulfur includes faster rate of particles sedimentation or chemical instability where separations of chemical phases occurred due to inhomogeneity. This consequently affects the final goods produced from them. A poor sulfur dispersion quality may affect vulcanization and crosslinking performance that will subsequently alter the final rubber goods performance in turn. Stable particle dispersions are often formed by adjusting the suspension's ionic strength and pH or by surface modification themselves by mechanical means (Inkyo et al., 2006). Thus, process selection and parameters play a great role in producing homogenous chemical dispersions such as sulfur. According to Wręczycki, Bieliński, & Anyszka (2018), crosslinks in the rubber network are distributed rather randomly and their inhomogeneity may be the result of poor dispersion of curatives such as sulfur during the preparation of sulfur dispersion from the raw material mixes to the subsequent processes that follow, or rather issues related to latex compounding process.

Thus, sulfur dispersion's mixing and grinding processes have been selected in this research, and these two processes' parameters optimization for sulfur dispersion were performed in order to revise and create a milestone for the full utilization and fine-tuning the optimized way in improving the quality and performance of the sulfur itself. Besides that, it will benefit the chemical dispersion manufacturing industry as the product defects can be minimized and as such, the sulfur consumer can manufacture rubber goods at relatively better performance. Additionally, no study has been conducted in optimizing the process conditions of sulfur dispersion in mixing and grinding processes in large scale. Ideally, this study will also benefit the end users of the rubber products to gain confidence from them and avoid the usage of second grade, rejected, or damaged rubber goods.

1.3 IMPORTANCE OF STUDY

The importance of this study is to enhance the raw chemicals mixing and grinding process of sulfur dispersion in a chemical plant by optimization study for both the processes. Sulfur in its dispersion form mainly acts as one of the contributing agents especially in rubber production, during the latex compounding procedure by introducing vulcanization.

Continuous advancements and researches were done in order to develop good rubber end-products. Thus, this study was done to contribute in improvements not only for the rubber products, but as well towards other industrial applications that uses sulfur as their main consumable or major raw material. Originating from the sulfur dispersion itself, this process enhancement study is hoped to benefit most sulfur-user industries, especially the producer/manufacturer which is the chemical dispersion plant itself in terms of quality improvement, process enhancement as well as waste elimination.

1.4 OBJECTIVES

The present study is mainly to develop to achieve the objectives as mentioned below:

- 1. To determine the optimum parameters for sulfur dispersion prepared via masterbatch mixing in 4 MT mixing tank in terms of its particle size distribution, viscosity, pH, and TSC.
- 2. To determine the optimum parameters for sulfur dispersion grinded in 60-Litre grinding mill in terms of its particle size distribution, viscosity, pH, and TSC.
- To analyze the morphology of sulfur dispersed in rubber film using SEM/EDX from selected sulfur dispersion sets of Objective 2.

1.5 SCOPES OF STUDY

For this research, the scopes as mentioned below have been identified:

- Design of Taguchi's L₉ orthogonal array for sulfur dispersion's mixing tank optimization with 2 factors (mixing speed and mixing time) and 3 levels for each factors (800, 600 and 400 rpm for mixing speed; with 30, 20 and 10 minutes for mixing time).
- Design of Taguchi's L₉ orthogonal array for sulfur dispersion's grinding mill optimization with 2 factors (motor speed and pump rate) and 3 levels for each factors (800, 700 and 600 rpm for motor speed; with 750, 550 and 350 L/min for pump rate).
- 3. The output of the mixing process was measured in terms of sulfur dispersion's chemical specifications as follows:
 - i. particle size distribution, D90,

- ii. viscosity,iii. pH, andiv. TSC.
- 4. The optimized sulfur dispersion mixing process was next chosen for its grinding mill optimization.
- Similarly, the output of the grinding process was measured in terms of sulfur dispersion's chemical specifications as follows:
 - v. particle size distribution, D90,
 - vi. viscosity,
 - vii. pH, and
 - viii. TSC.
- 6. After the grinding mill step, evaluation on instability index of various selected sulfur dispersions sets (after grinding mill) with index value results close to "0" as most stable and close to "1" as least stable sulfur dispersion; was performed.
- 7. Apart from that, rubber films were casted using sulfur dispersions obtained from the steps above and characterized for its morphology using SEM/EDX to analyze the elemental compositions on the rubber films' surface.

1.6 THESIS ORGANIZATION

This thesis mainly consists of five chapters. Firstly, Chapter One was initiated with a brief description of the research's background which include descriptions on goal of organizations or manufacturers to produce good quality products with optimizations. Separately, description of the product chosen (sulfur dispersion) was also briefed to connect with optimization. Apart from that, details on problem statement, research objectives together with their scopes and importance of the research were also listed and described for readers' attention.

Subsequently, Chapter Two further thoroughly detailed on the elements presented in the research. As such, each detail on sulfur and its dispersion form were reviewed and detailed in the subsequent sub-chapter. Taguchi's approach as the optimization tool was also detailed in this chapter to provide a clear basis for a better understanding of the readers before proceed to the main focus of the research, which is optimizing process parameters of sulfur dispersion product.

Chapter Three presented the flowchart of the research methodology that fulfills the three objectives as mentioned in Chapter One. The flowchart would be helpful to provide the idea on the flow of the research in general. This chapter also revealed on the chosen materials, formulation, and parameters in terms of the design of Taguchi's L₉ orthogonal array for mixing process and grinding mill process of the sulfur dispersion, with chosen sulfur dispersion's specifications (factors, levels and responses).

Chapter Four in this thesis discussed thoroughly on the compiled, tabulated, and analyzed results for both optimization studies (mixing process parameters and grinding mill parameters). Generally, the selected or optimized sulfur dispersion's mixing process was chosen for optimization of the next process step – grinding mill parameter optimization. Consequently, means and S/N ratios were calculated for each run and the results were analyzed using ANOVA table that measures p-value to indicate the significance of each of the factors chosen. Additionally, morphology of the sulfur sets on the surface of rubber film casted was also observed, analyzed and discussed in this chapter.

Lastly, Chapter Five summarized and concluded the overall research and the results' significance. Apart from that, recommendations on further improvements on the research work were also outlined.

CHAPTER 2

LITERATURE REVIEW

2.1 BACKGROUND OF SULFUR

The Earth is richly equipped with various chemical elements in different regions in land and in the sea. As the elements naturally exist, their usage is exceptionally important for living things. Examples of elements include carbon, oxygen, zinc, magnesium, calcium and so much more. Sulfur (S) is one of the elements abundantly present in bulk on the Earth, both naturally and by chemical means. Its usage is exceptionally important as well especially in different industrial applications.

Today, in some regions of the world, sulfur (S) (in its powder or derived form) or interchangeably spelled as sulphur, has been widely manufactured and used in many industrial applications (Blight, Currell, Nash, Scott, & Stillo, 1978; Nehb & Vydra, 2012). U.S was the largest producer of elemental sulfur in the world, followed by Canada, in 2013. Apparently, China shot up its sulfur production until the year 2015, then continued throughout until 2018 and became the world's largest manufacturer of sulfur (Kutney, 2013). Figure 2.1 by the U.S. Geological Survey, (2019) shows the graph of sulfur production from the year 2013 to 2018, by country.