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(54) Title: RUBBER TREATMENT METHOD

(57) Abstract: A process and resulting product is provided in which vulcanised rubber crumb has selected chemical bonds broken and sulphur removal by biotreatment with hydrophobic bacteria, such as mycolic acid containing actinomycete bacteria or 'mycolata', without significant degradation of the hydrocarbon polymer. The product obtained from the use of these bacteria may be reprocessed alone or in blends with virgin rubber and revulcanised to yield products of much higher quality than conventional reclaimed rubber materials.

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“Rubber Treatment Method”

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The present invention relates to a method of treating rubber. In particular, it relates to a method of treating vulcanised rubber in order that the rubber may be reprocessed or recycled.

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Background to the Invention

The largest single application for rubber is in vehicle tyre. The principal rubbers used are the hydrocarbon polymers Natural Rubber (NR), Styrene Butadiene Rubbers (SBR) and Polybutadiene Rubbers (BR). During processing the polymer molecules are vulcanised i.e. cross-linked by sulphur atoms. The formation of cross-links enhances the mechanical properties of the rubber but renders it unsuitable for easy processing. Throughout this application, vulcanised rubber is taken to mean rubber cross-linked by sulphur.

Waste rubber materials, such as vehicle tyres, present a significant environmental problem.

1 Currently, the European Union scraps a total of nearly 2×10^6 tonnes of tyres
2 per year of which 23% are retreaded and 46% disposed to landfill. In the UK,
3 it is estimated that approximately 100000 tonnes of tyres are disposed of each
4 year, mainly in landfill sites. In the USA, 300 million tyres are disposed to
5 landfill per annum. In New York State alone, 12 million tyres are discarded
6 per annum, which represents in excess of three million barrels of oil in energy
7 equivalent being discarded.

8

9 The United Nations and EU have warned that waste rubber is becoming a
10 significant environmental problem worldwide. The EC Landfill Directive
11 (1999/31/EC) has advocated the banning of disposal to landfill by 2003 for
12 whole tyres and 2006 for shredded tyres.

13

14 Currently, alternative means of disposal of waste rubber, in particular waste
15 tyres, include various recycling methods and burning of e.g. in cement kilns.
16 However, burning of rubber materials such as tyres can produce significant
17 pollutants including dioxins.

18 Life cycle analysis shows that only a small fraction of the energy used in
19 manufacturing tyres is recovered on combustion.

20 Conventional recycling methods include mechanical, thermo-mechanical,
21 cryomechanical, microwave and ultrasonic methods. Chemical recycling
22 methods include reclamation using organic disulphides, mercaptans and
23 inorganic compounds. However, rubber produced using these methods has
24 poor mechanical properties. There is also pyrolysis of waste rubber to oils and
25 carbon black. Alternative methods of recycling include employing reclaimed
26 rubber that is blended with Low Density Polyethylene (LDPE) to produce
27 mixed polymer elastomeric material. However, rubber produced using these
28 methods has poor mechanical properties.

29 Rubber crumb is also used in roads, playparks, running tracks and equestrian
30 surfaces. However none of these applications come close to using the large
31 quantities of waste rubber available.

32

1 A recent biotechnological approach to recycling rubber involves elastomer
2 recycling to the viscoelastic state by removal of sulphur by the thermophilic
3 bacterium *Sulfolobus*. However, the use of a thermophile at temperatures in
4 the region of 70° carries a large energy cost penalty and generates highly
5 acidic environments with production of sulphuric acid which can lead to
6 reprocessing problems. DE04042009 describes the surface treatment of rubber
7 crumb by suspensions of chemolithotrophic Sulphur-oxidising bacteria
8 allowing revulcanisation to take place when mixed with new (virgin) rubber
9 stock and devulcanisation of comminuted rubber scrap by similar suspensions
10 of chemolithotrophs to produce viscoelastic rubber and sulphuric acid.
11 However, the presence of sulphuric acid in the reaction mixture would be
12 detrimental to revulcanisation and to the quality of any reprocessed rubber.

13

14 As current methods of disposal of waste rubber are environmentally
15 unacceptable and conventional methods of recycling produce low quality
16 rubber materials, there remains a need for a method of recycling rubber
17 materials which produces rubber of high quality and reduces environmentally
18 unacceptable consequences.

19

20 **Summary of the Invention**

21

22 The present inventors have surprisingly discovered that hydrophobic bacteria
23 for example mycolic acid containing actinomycete bacteria or 'mycolata' can
24 efficiently devulcanise waste rubber products without causing significant
25 degradation of the unsaturated hydrocarbon polymer forming the rubber
26 chains. The rubber recycled using these bacteria may be revulcanised to
27 produce rubber material of a much higher quality than conventional recycled
28 rubber materials.

29

30 Accordingly, in a first aspect of the present invention, there is provided a
31 method of devulcanising rubber comprising, providing a vulcanised rubber
32 substrate, exposing the vulcanised rubber substrate to mycolata bacteria, and

1 allowing the mycolata bacteria to break down C-S and S-S bonds in the
2 vulcanised rubber substrate to produce a devulcanised rubber.

3

4 Throughout this specification, reference to devulcanisation refers to the
5 breaking of C-S and S-S bonds between rubber molecules. Reference to
6 "devulcanised rubber" refers to rubber in which C-S and S-S bonds have been
7 broken.

8

9 Preferably, devulcanised rubber produced according to the method of the
10 invention has less than 90%, preferably less than 85%, more preferably less
11 than 80%, even more preferably less than 75%, yet more preferably less than
12 70%, even more preferably less than 65%, most preferably less than 60% of
13 the C-S and S-S bonds originally present in the "vulcanised" form prior to
14 treatment using the method of the invention. This may be assessed using
15 conventional means of measuring total sulphur content.

16

17 Preferably, the method of the invention removes at least some sulphur from
18 the rubber substrate. Thus, preferably, devulcanised rubber produced
19 according to the method of the invention has less than 90%, preferably less
20 than 85%, more preferably less than 80%, even more preferably less than
21 75%, yet more preferably less than 70%, even more preferably less than 65%,
22 most preferably less than 60% of the total sulphur content of the "vulcanised"
23 form prior to treatment using the method of the invention.

24

25 Preferably, the devulcanised rubber product produced using the method of the
26 first aspect of the invention is of sufficient quality to be capable of
27 reprocessing and revulcanisation and reprocessing to a new rubber product
28 e.g. rubber tyre, without the need for addition of virgin rubber. However, the
29 recycled rubber is capable of being blended and reprocessed with virgin
30 rubbers in all proportions. Preferably, a tyre produced using such a recycled
31 rubber would meet or exceed the relevant safety and quality standard set for
32 vehicle tyres by UK, EU or US regulatory authorities.

1
2 According to a second aspect of the present invention, there is provided a
3 method of recycling a vulcanised rubber comprising devulcanising the rubber
4 according to the first aspect of the invention, and reprocessing the
5 devulcanised rubber. Reprocessing may include blending with virgin rubber
6 and/or may include addition of any further ingredients prior to revulcanisation
7 to yield a high quality rubber product. The reprocessing temperature will
8 generally be sufficient to kill and/or destroy most, preferably all, of the
9 bacteria. Alternatively the devulcanising bacteria can be removed from the
10 rubber by alkali washing prior to further processing.

11
12 Examples of mycolata bacteria which may be used in the present invention
13 include, but are not limited to, members of the genera *Corynebacterium*,
14 *Rhodococcus*, *Nocardia*, *Gordonia*, *Tsukamurella*, *Dietzia* and
15 *Mycobacterium*. In a preferred embodiment, the bacterium is of the genus
16 *Gordonia*. In a particularly preferred embodiment, the bacteria is the
17 *Gordonia desulfuricans* strain SG213E, samples of which were deposited
18 with the National Collections of Industrial and Marine Bacteria Ltd.
19 (NCIMB), 23 St Machar Drive, Aberdeen, Scotland AB24 3RY on 28
20 February 2003 and also on 29 July 1996 under accession no NCIMB 40816.

21
22 The methods of the invention may be used on any type of vulcanised rubber,
23 in particular waste rubber, such as rubbers used in vehicle tyres. Rubbers for
24 use in the invention may be natural or synthetic or a mixture thereof.
25 Synthetic rubbers include but are not limited to Styrene Butadiene Rubbers
26 (SBR) and Polybutadiene Rubbers (BR). Examples of structural formulae for
27 vulcanised natural rubber, synthetic Styrene Butadiene rubber
28 and the model compound allyl disulphide are shown in Figure 1.

29
30 In order to maximise the surface area of rubber material for reaction with the
31 bacteria, the rubber material is preferably provided in particulate form e.g.
32 such as in the form of rubber crumb, the preparation of which is known to the

1 person skilled in the art. Preferred particle, e.g. crumb, sizes are in the range
2 0.1mm to 15mm, for example 1mm to 15mm. Such particles, e.g. crumbs,
3 may be formed using any method known in the art, for example mechanical
4 and/or cryogenic grinding.

5
6 Moreover, the present inventors have found that the efficiency of the methods
7 of the invention and the quality of the devulcanised rubber produced using the
8 methods of the invention may be enhanced by carrying out the reaction in the
9 presence of rubber processing oils such as hydrocarbon oils, and similar
10 materials which contain long hydrocarbon sequences in the molecular
11 structure e.g. stearic acid. In a preferred embodiment the rubber processing
12 oil is stearic acid. In a further preferred embodiment, the rubber processing oil
13 is hexadecane.

14
15 Without being limited to a particular mechanism it is believed that by coating
16 the rubber particles with such oils, or similar materials, the surface area on
17 which the bacteria may act is increased and access to the C-S and S-S bonds is
18 improved, enabling more efficient breaking of the C-S and S-S bonds and
19 preferably removal of sulphur to leave a devulcanised rubber. Moreover, by
20 coating the rubber particles in such oil, or similar materials, the bacteria may
21 use the oil as a Carbon source for growth instead of using the rubber
22 hydrocarbon polymer, thus further limiting the degradation of the rubber
23 hydrocarbon chains. Accordingly, in particularly preferred embodiments of
24 the invention, the rubber material e.g. in the form of rubber crumb, is reacted
25 with bacteria in the presence of oils, or similar materials, which can act as
26 carbon sources for the devulcanising bacteria. Suitable oils include but are not
27 limited to mineral oils such as paraffinic, naphthenic, aromatic and white oils.
28 Particularly preferred for use in the methods of the invention are oils which
29 allow swelling of the rubber with greater access to sulphur cross-links. In
30 addition, the metabolism of such rubber processing oils which may be used by
31 the bacteria in the methods may result in the production of surface active

1 agents (surfactants) which further enable access of the bacteria to the sulphide
2 bridges.

3
4 Although any suitable temperature range may be used in the methods of the
5 invention, the temperature being determined principally by the optimum
6 temperature required for activity of the particular bacteria being used, in
7 preferred embodiments the bacteria are active in breaking the C-S bonds and
8 S-S bonds at mesophilic temperature ranges, e.g. in the range 15-40°C,
9 preferably in the range 20-35°C. The use of bacteria which devulcanise rubber
10 products at room temperature enables gentler treatment of the rubber products
11 compared to conventional treatments to reclaim rubber, many of which
12 require treatment at high temperatures. The use of mesophilic bacteria
13 enables the breakdown of C-S and S-S bonds between the unsaturated
14 "rubber" hydrocarbon polymer chains, which constitute the main rubber
15 chains, without degrading the hydrocarbon polymer.

16
17 In preferred embodiments, the method is carried out under conditions of pH in
18 the range pH5 to pH9, preferably in the range pH6 to pH8, most preferably at
19 around pH 7 i.e. in the range pH 6.5 to pH 7.5. Preferably, the method is
20 performed under aerobic conditions.

21
22 The methods of the invention may be carried out in any suitable reaction
23 vessel, including biopiles, preferably with means for controlling reaction
24 conditions. Various reaction conditions and factors may be modified in order
25 to control the rate and extent of bacterial devulcanisation according to the
26 method of the invention. For example, the rate and/or extent of bacterial
27 devulcanisation of rubber product may be controlled by controlling one or
28 more of oxygen tension, redox potential, temperature, process oil
29 concentration, mixing speed during devulcanisation, and/or physical and/or
30 chemical treatment of the rubber substrate e.g. rubber crumb prior to
31 devulcanisation according to the method of the invention.

32

1 The rubber material may thus be pre-treated prior to reaction with the
2 bacteria. Such pre-treatments may include method steps to at least partially
3 remove textile fibres, metal beads and other constituents in the vulcanised
4 rubber as well as chemical and/or mechanical treatments.

5
6 Preferably, during reaction, the reaction mixture of rubber and bacteria are
7 agitated together, e.g. using a rotating drum. Mixing speed may be adjusted to
8 provide control of the rate of reaction.

9
10 In preferred embodiments of the invention, the method is carried out under
11 conditions of undetectable or very low sulphur concentration e.g. less than
12 0.025%, preferably less than 0.01% sulphur such that the bacteria degrade the
13 sulphur bonds between the rubber molecules to utilise the sulphur in the
14 vulcanised rubber causing devulcanisation. Preferably the method is carried
15 out whereby all trace elements necessary for the growth of the bacteria are
16 present except for sulphur,

17
18 The incubation time of bacteria with the rubber substrate according to the
19 method of the invention may be adjusted dependent on a number of factors
20 including particular bacteria used, temperature of reaction, size of rubber
21 substrate particles used, presence and nature of carbon support. Typically, the
22 rubber substrate will be incubated with the bacteria for an incubation time in
23 the range 1 to 96 hours or longer.

24
25 In preferred embodiments, the oxygen tension may be in the range 0.5-20
26 mg/L, for example 2-20 mg/L. More preferably, the oxygen tension is in the
27 range 0.5-9.0 mg/L. Most preferably the oxygen tension is 4-8 mg/L. The
28 oxygen tension may however be higher depending on the system internal
29 pressure.

30
31 Following treatment, the devulcanised rubber may be washed and filtered in
32 order to remove any residual bacteria. Additionally or alternatively, any live

1 residual bacteria may be killed by reprocessing the rubber at high
2 temperatures. For example, reprocessing of the devulcanised rubber will
3 typically take place at temperatures in excess of 100°C, for example 150°C,
4 which will kill bacteria such as the mycolata which are preferred for use in the
5 methods of the invention.

6

7 In a further aspect of the invention there is provided devulcanised rubber
8 produced according to the methods of the invention.

9

10 Devulcanised rubber of the invention and made according to the present
11 invention has superior properties to recycled rubber produced according to
12 conventional methods and may be revulcanised and used in the production of
13 new rubber products, for example in the production of tyres. The quality of
14 rubber produced by the methods of the invention may be tested using
15 conventional rubber quality criteria known to those versed in the art. Such
16 criteria include plasticity (e.g. ASTM Standard D1646), scorch (e.g. ASTM
17 Standard D1646), minimum viscosity (e.g. ASTM Standard D1646), shore
18 hardness (e.g. ASTM Standard D2240), modulus of elongation at 300% and
19 100% (e.g. ASTM Standard D412, test method A), elongation at break (e.g.
20 ASTM Standard D412, test method A), energy at break (e.g. ASTM Standard
21 D412, test method A as set forth in units of MPa), G' (e.g. ASTM Standard
22 D2221) and Tan Delta (hysteresis measured in accordance with ASTM
23 Standard D2231).

24

25 The invention will now be exemplified with reference to the following non-
26 limiting description and the accompanying Figure 1, which shows structural
27 formulae of some vulcanised rubber molecules.

28

29 Examples

30

31 Using old tyres, rubber crumb is prepared and treated with process oil such as
32 one or more mineral oils such as paraffinic, naphthenic, aromatic or white oil

1 in a reaction vessel. A bacterial suspension of *Gordonia desulfuricans* strain
2 SG213E (NCIMB 40816 or as deposited with NCIMB on 28 February 2003)
3 is mixed with the rubber crumb and process oil at room temperature (20-
4 30°C) for a period of 2-12 days, but preferably 4-7 days. The presence of the
5 oil swells the rubber crumbs, allowing enhanced access of the bacteria to C-S
6 and S-S bonds, while protecting the rubber hydrocarbon chains from
7 degradation by acting as a C source for the bacteria. Temperature, aeration,
8 crumb mass to process oil ratio, and other physico-chemical factors are
9 controlled to optimise reaction conditions.

10

11 After reaction, the devulcanised rubber is recovered from the reaction vessel,
12 filtered and washed and tested for extent of devulcanisation and quality of
13 rubber product using tests known in the art. Suitable properties of the rubber
14 product which may be tested include one or more of tensile strength, modulus,
15 hardness, tear resistance and solvent swelling of the recovered rubber. Tests
16 for the appearance of inorganic sulphur in the process effluent or liquid
17 residue may also be carried out,
18 as well as total sulphur content of the rubber.

19

20 In addition, periodically during the reaction period, samples may be removed
21 from the reaction vessel and tested for extent of devulcanisation and product
22 quality using one or more of such tests.

23

24 **Example 1**

25 50g of 12 mesh rubber crumb derived from truck tyres was rotated in a
26 horizontal cylindrical vessel with 100 cm³ of aqueous medium containing
27 essential minerals, between 0.1 and 3 cm³ of hexadecane and 500µl innoculum
28 of a late logarithmic growth phase culture of *Gordonia desulfuricans*
29 (NCIMB 40816) grown in a similar medium supplemented with
30 benzothiophene as a sulphur source.

31 Typical aqueous medium composition in gdm⁻³

32 Na₂HPO₄ 4.33g, KH₂PO₄ 2.65g, NH₄Cl 2g,

1 MgCl₂.6H₂O 0.64g, CaCl₂.2H₂O 33mg, ZnCl₂ 2.6 mg,
2 FeCl₂.4H₂O 2.6mg, EDTA 1.25mg, MnCl₂.4H₂O 1mg, CuCl₂.2H₂O 0.15mg,
3 Co(NO₃)₂.6H₂O 0.125mg,
4 Na₂B₄O₇.10H₂O 0.1mg, (NH₄)₆Mo₇O₂₄.4H₂O 0.09mg.

5 The medium was changed when maximum growth of the bacteria was
6 observed. This change could be repeated several times. The percentage
7 reduction of total sulphur content in the rubber samples after treatment varied
8 between 23% and 35%.

9 When blended with virgin rubber stock at a loading of 40% by weight and
10 revulcanised, the treated crumb samples showed good tensile strength
11 substantially higher than similar vulcanised blends containing untreated
12 crumb. Eg

13 Blend with 40% treated crumb, 20.9 MPa

14 Blend with 40% untreated crumb, 17.1 Mpa

15

16 Example 2

17 The same process was carried out as in Example 1 but with hexadecane is
18 replaced by glycerol. In contrast to example 1 when hexadecane is replaced
19 by glycerol as a carbon support there is no significant reduction in the total
20 sulphur content of the rubber.

21

22 Example 3

23 The same process was carried out as in Example 1 but with *Gordonia*
24 *desulfuricans* replaced by a *Rhodococcus erythropolis* strain DT10 . In
25 contrast to example 1, the reduction in total sulphur content of the rubber was
26 substantially lower at 13%.

27

28 Example 4

29 The same process was carried out as in Example 1 but with *Gordonia*
30 *desulfuricans* replaced by the *Rhodococcus erythropolis* strain used in
31 example 3 and the hexadecane replaced by glycerol. In contrast to example 1
32 there was no significant reduction in total sulphur content of the rubber.

1

2 Example 5

3 The same process was carried out as in Example 1 but with *Gordonia*
4 *desulfuricans* is replaced by a further *Rhodococcus erythropolis* strain. In
5 contrast to example 1 when *Gordonia desulfuricans* was replaced by a further
6 *Rhodococcus erythropolis* strain DT05, the reduction in total sulphur content
7 of the rubber was 11%.

8

9 Example 6

10 The same process was carried out as in Example 1 but with *Gordonia*
11 *desulfuricans* replaced by the same strain of *Rhodococcus erythropolis* as
12 used in example 5 and hexadecane replaced by glycerol. In contrast to
13 example 1 the reduction in total sulphur content of the rubber was only 9%.

14

15 All documents referred to in this specification are herein incorporated by
16 reference. Various modifications and variations to the described embodiments
17 of the inventions will be apparent to those skilled in the art without departing
18 from the scope and spirit of the invention. Although the invention has been
19 described in connection with specific preferred embodiments, it should be
20 understood that the invention as claimed should not be unduly limited to such
21 specific embodiments. Indeed, various modifications of the described modes
22 of carrying out the invention which are obvious to those skilled in the art are
23 intended to be covered by the present invention.

1 Claims

2

3 1. A method of devulcanising rubber comprising
4 providing a vulcanised rubber substrate,
5 exposing the vulcanised rubber substrate to
6 mycolata bacteria, and allowing the mycolata
7 bacteria to break down C-S and S-S bonds in
8 the vulcanised rubber substrate to produce a
9 devulcanised rubber.

10

11 2. The method according to claim 1, wherein the
12 devulcanised rubber has less than 95% of the
13 C-S and S-S bonds originally present in the
14 vulcanised rubber substrate.

15

16 3. The method according to claim 2, wherein the
17 devulcanised rubber has less than 70% of the
18 C-S and S-S bonds present in the vulcanised
19 rubber substrate.

20

21 4. The method according to any one of the
22 preceding claims, wherein the devulcanised
23 rubber has less than 90% of the total
24 sulphur content of the the vulcanised rubber
25 substrate.

26

27 5. The method according to any one of the
28 preceding claims, wherein the mycolata
29 bacteria comprise bacteria of at least one
30 of the genera *Corynebacterium*, *Rhodococcus*,
31 *Nocardia*, *Gordonia*, *Tsukamurella*, *Dietzia* or

1 *Mycobacterium*.

2

3 6. The method according to claim 5, wherein the
4 mycolata bacteria comprise bacteria are of
5 the genus *Gordonia*.

6

7 7. The method according to claim 6, wherein the
8 *Gordonia* bacteria are *Gordonia desulfuricans*

9

10 8. The method according to claim 7, wherein the
11 *Gordonia* bacteria are *Gordonia desulfuricans*
12 strain SG213E (accessionno: NCIMB 40816).

13

14 9. The method according to claim 5, wherein the
15 mycolata bacteria comprise bacteria of the
16 genus *Rhodococcus*.

17

18 10. The method according to claim 9, wherein the
19 *Rhodococcus* bacteria are *Rhodococcus*
20 *erythropolis*.

21

22 11. The method according to any one of the
23 preceding claims, wherein the rubber
24 substrate is in particulate form, wherein
25 the particles have a cross section in the
26 range 0.1mm to 15mm.

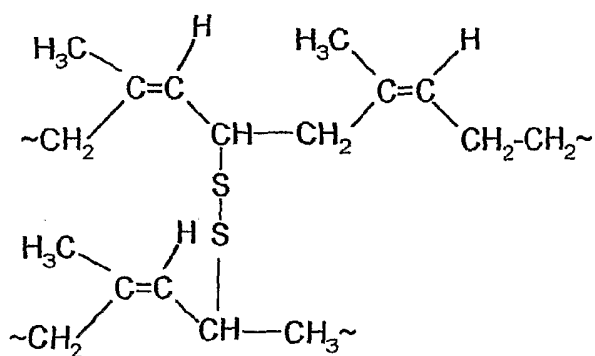
27

28 12. The method according to any one of the
29 preceding claims wherein the rubber
30 substrate is exposed to the mycolata
31 bacteria in the presence of one or more

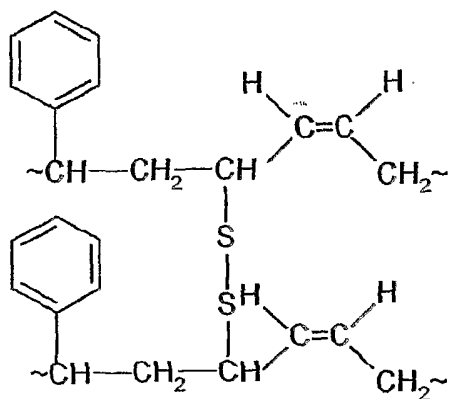
- 1 processing oils.
- 2
- 3 13. The method according to claim 12, wherein
- 4 the processing oil is stearic acid or
- 5 hexadecane.
- 6
- 7 14. The method according to any one of the
- 8 preceding claims wherein the method is
- 9 performed at a temperature in the range 15-
- 10 40°C.
- 11
- 12 15. The method according to any one of the
- 13 preceding claims wherein the method is
- 14 performed under conditions of less than
- 15 0.025% sulphur.
- 16
- 17 16. The method according to any one of the
- 18 preceding claims wherein the method is
- 19 performed under conditions of oxygen tension
- 20 in the range 0.5-9.0 mg/L.
- 21
- 22 17. A method of recycling vulcanised rubber
- 23 comprising devulcanising the vulcanised
- 24 rubber using the method of any one of the
- 25 preceding claims and revulcanising the
- 26 devulcanised rubber product to produce a
- 27 recycled rubber product.
- 28
- 29 18. The method according to claim 17, wherein
- 30 the devulcanised rubber is reprocessed at a
- 31 temperature of greater than 100°C.
- 32

- 1 19. The method according to claim 17 or 18
2 comprising the step of forming a tyre using
3 the recycled rubber product.
4
- 5 20. A devulcanised rubber produced according to
6 the method of any one of claims 1 to 16.
7
- 8 21. A recycled rubber product produced according
9 to the method of any one of claims 17 to 19.
10
- 11 22. A tyre comprising recycled rubber product
12 produced using the method of claim 17 or 18.
13
- 14 23. A tyre produced according to the method of
15 claim 19.
16

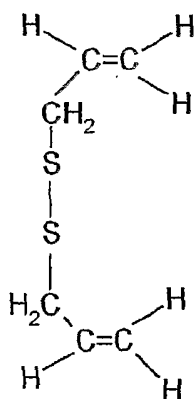
Figure 1

a) Natural Rubber (*cis*-1,4-polyisoprene)

b) Synthetic Styrene Butadiene Rubber (SBR)



c) Model Compound- Allyl disulphide



Bond Energies (kJ mole⁻¹): C=C, 598; C-C, 349; C-S, 272, S-S, 266; C-H, 372